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Contract Scientific Authority:
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Contract Report
DRDC Suffield CR 2007-240
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Contract Report

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PRELIMINARY EVALUATION OF THE PROPERTIES OF DYNAMICALLY VULCANISED THERMOPLASTIC RUBBERS

Contract No: W7702-03R978/001/EDM

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Abstract

The report presents work on five types of dynamically vulcanized thermoplastic rubber blends made from chlorobutyl and nitrile rubbers and polypropylene, nylon 12, and PPT resins.

The project objective was to establish a correlation between blend composition and properties such as microstructure swelling index, tensile strength, elongation at break, hardness, and resistance to penetration/reemission of chemical warfare agents.

The method of blend preparation and materials testing was described.

Results were reviewed and conclusions were drawn.

The results confirm that dynamic vulcanization can produce a variety of rubber plastic blends, many with elastomeric properties. Both rubber and plastic phases seems to be affected during the dynamic vulcanization process. Under exposure to solvents, both vulcanized rubber and rubber/plastic blends underwent a rapid approach to equilibrium swelling. It was found that swelling index values of blends were significantly less that the expected "theoretical" values, based strictly on composition in the blend. This was attributed to a caging effect of the thermoplastic phase on the rubber phase at the higher thermoplastic composition.

In blends of rubber and thermoplastic, the minimum elongation at break seems to be reached at phase inversion. With respect to re-emission values for the five blends systems studied, from best to worst, the order is PA/CIIR > PP/CIIR > PA/NBR > PP/NBR > PBT/NBR.

Selected results were presented during the IUPAC International Polymer Congress in Paris (July 4-8, 2004). Overheads from the presentation are enclosed with report.

Keywords

Polybutyleneterephthalate, polypropylene, polyamide 12, chlorobutyl rubber, nitrile rubber, dynamic vulcanization, swelling index, microstructure, tensile strength, elongation at break, hardness, resistance to penetration, reemission.

INTRODUCTION

Previous reviews in the defence literature of chemical agent and POL resistance of thermoset and homopolymer materials indicated the potential to use chemically agent resistant thermoplastic and elastomeric materials to produce novel thermoplastic elastomers (TPE) as blends. When prepared by dynamic vulcanisation, the morphology of these materials is such that two separate microscopic phases exist, one consisting of a suitable thermoplastic phase, and the other composed of an elastomeric material. These materials have the property of heat mouldability and process ability at high processing temperatures, while having useful elastomeric performance at working temperatures.

In the course of contract W7702-02R905/001/EDM the properties of a series of polyamide/chlorobutyl rubber blends prepared by dynamic vulcanisation were correlated with composition of polyamide in the blends (15 – 40% polyamide). In plots of swelling index versus percentage polyamide of these blends, it was observed that the swelling index of each blend combination was significantly less than the calculated expected swelling index based on the ratio of plastic to rubber. Furthermore, the plot underwent a unique change in slope at a composition of approximately 25% polyamide. By reference to scanning electron microscopy, it was suspected that this composition roughly corresponded to a polyamide/rubber ratio where the continuous phase in the system became dominated by the uncured thermoplastic phase. In addition, it has been observed that at this approximate composition the values of elongation at break reach a minimum, and values of re-emission of warfare agents undergo a change in slope versus composition.

A significant consequence of the above results is that TPE blends made from polyamide and chlorobutyl rubber may have improved chemical resistance to solvent attack in comparison to the expected results, especially for blends with polyamide compositions exceeding approximately 25%. Furthermore, swelling index of the thermoset rubber phase in these blends may be a useful indicator of changes in the morphology of thermoplastic/rubber compositions, and also a predictor of mechanical and barrier properties changes with composition.

Results presented in this report were the basis of the presentation entitled “Solvent Resistance and Mechanical Properties in Thermoplastic Elasotomer Blends Prepared by Dynamic Vulcanization” presented during the IUPAC International Polymer Congress in Paris (July 4-8, 2004). Overheads from the presentation are presented in the appendix to this report.

OBJECTIVE

The objective of the proposed blending program was to confirm whether improvements in solvent resistance for polyamide/chlorobutyl bends (as measured by swelling index) apply to a variety of thermoplastic/rubber combinations. Information of this nature is likely to be of interest in the design of protective gear for warfare agents.

Correlations between composition and physical properties of TPE materials made by dynamic vulcanisation would be sought. The blends would be made prepared using three different thermoplastic resins - Polyamide 12 (PA), polypropylene (PP), and polybutylene terephthalate (PBT), and two different rubbers - chlorobutyl rubber (CIIR) and nitrile rubber (NBR).

Specifically, the goal of this contract was to extend the correlation between composition and properties such as swelling index, tensile strength, elongation at break, hardness, and resistance to penetration/re-emission of chemical warfare agents to five resin/rubber combinations – namely PA/CIIR, PP/CIIR, PA/NBR, PP/NBR, and PBT/NBR.

The data generated in this report became the basis for a presentation to the World Polymer Conference (MACRO 2004), Paris, France titled "Solvent Resistance and Mechanical Properties in Thermoplastic Elastomer Blends Prepared by Dynamic Vulcanization", July 6, 2004 by J.D. Van Dyke, M. Gnatowski, and A. Burczyk (copy attached).

SCOPE OF WORK CONDUCTED

a. Dynamically vulcanised blends of thermoplastic resin and rubber were made at up to seven different ratios (15-50% thermoplastic). In addition, 100% rubber samples utilizing the same curing system as in the blends were mixed and cured in a press. Samples of pure resins were masticated and prepared for testing in the same manner as for the TPE blends. A ZDEDC/Zn curing system was used for chlorobutyl rubber blends and a bismaleinide (or bismaleimide-peroxide) curing system was used for NBR.

b. Evaluations were undertaken for swelling index, percentage insolubles, tensile strength, and elongation at break, as well as hardness. Specimens were also made for evaluation of warfare agent penetration and re-emission at DRDC Suffield.

c. Part of the project involved experimentation with blend preparation, because there was no literature support or other data describing the details of the required blending conditions.

d. Originally it had been anticipated that PBT/CIIR blends would be included in the testing. However, during blending of these two components, decomposition took place making all PBT/CIIR and PBT/IIR blends unsuccessful.

e. DSC and SEM were used to determine whether the thermoplastic and rubber phases were separate in the mixtures, and evaluate several blends to identify possible polymer interaction and degradation.

f. A research presentation of this body of work was made at the World Polymer Congress in Paris 2004, entitled "Effect of Composition on the Properties of a Variety of TPE Blends by Dynamic Vulcanization" by J.D. Van Dyke, Marek Gnatowski, and Andrew Burczyk.

EXPERIMENTAL PROCEDURES

1. Mixing Procedure

All blends were made in a 258 cm3 capacity 5 HP Plasticorder EPL-V5502 equipped with Prep Mixer type R.E.E.6 and type 808-2504/PSI/DTI Rheometer and temperature control (Brabender Instruments Inc., Hackensack, NJ).

For all dynamically-vulcanised blends, the plastic resin was first added at 30 rpm mixing speed to the Brabender Mixer at a target temperature of 190ºC, and allowed to melt for 2 minutes. The rubber was then added along with stearic acid, metal oxides, and wax, and the mixing speed was increased to 65 rpm. Mixing was continued for an additional 6 minutes. After a total mixing time of 8 minutes, the active curing agent was added and allowed to mix for 4.5 minutes (total mixing time of 12.5 minutes). The blend was then removed from the mixer and cooled.

All dynamic vulcanised blends used for injection moulding contained 0.8 phr Paracin 285 wax (on rubber).

Chlorobutyl rubber was masticated at 25 rpm and a target temperature of 60ºC for 3 minutes, after which stearic acid, zinc oxide and antioxidant were added, and mixing continued for an additional 2 minutes. MBTS and THTD were then added and mixing continued for 3 minutes. ZDEDC and Zn were then added, and mixing was complete after an additional 2 minutes. The total mixing time was 10 minutes. The curing levels (based on rubber) were identical to those used in the dynamic vulcanisation experiments. After blending was complete, the compound was compression moulded at 160ºC for 30 minutes.

Nitrile rubber formulations and mixing procedures varied depending on whether the blend was with polyamide, polypropylene, or polybutylene terephthalate. A blending procedure and timeline for nitrile rubbers is shown in Figure 1.

2. Testing and Characterization

a) Mechanical properties – In all blends the mechanical properties were tested on specimens prepared directly by injection moulding, (see Table 1B). A computerized Instron 4400 Universal Testing Machine was used to determine stress/strain characteristics on injection-moulded samples according to the American Society for Testing and Materials procedure D638 (ASTM D638).

b) Hardness values (Shore A and D) were determined by ASTM D2240 using a PTC Type A Durometer Gauge Model 306L or Type D Durometer Gauge Model 307L (PTC Instruments).

c) Swelling index and percentage insolubles – Swelling index and percentage insolubles were determined on moulded or pressed samples, ~1 cm square and 1.5 mm thick, that were immersed in hexane for 4 days to obtain equilibrium. The swelling index of a blend sample was determined by comparison of the weight of the swollen sample to its weight after drying to constant weight. The percentage insolubles of the sample in the solvent was determined by comparison of the dry to original weight.

d) Differential Scanning Calorimetry - For DSC, injection moulded samples of blends were tested and compared to non-processed mixtures of rubber and plastic of similar combination. Analysis was conducted on a Perkin Elmer DSC-7 instrument (Perkin Elmer Cetus Instruments, Norwalk, CT) according to ASTM D 3417. The melting temperature and the enthalpy of fusion for the plastic phase were recorded for each sample. If multiple peaks occurred, the melting temperature refers to the highest (second) peak.

e) Scanning electron microscopy – SEM was conducted using a variable pressure LEO 1455VP microscope (Meridian Scientific Services, Stittsville, ON, Canada). Specimens were prepared by cutting with a sharp blade or by fracture after cooling in liquid nitrogen. All samples were stained with osmium tetroxide, but staining was particularly effective in samples containing nitrile rubber. The blends were mounted on aluminum stubs with carbon paint, and examined without coating at low pressure using a Robinson Backscatter Detector.

TABLES AND FIGURES ATTACHED TO THIS REPORT

TABLE 1 A: Materials Used in Blending Study

Product	**Grade**	**Supplier**
Chlorobutyl Rubber (CIIR)	1068	Exxon
Nitrile Rubber (NBR)	Acrylonitrile-Butadiene Rubber - Krynac 3345C	Bayer
Polyamide (PA)	Nylon 12 (L16)	EMS-American Grilon
Polypropylene (PP)	Profax 6524	Basell
Polybutylene Terephthalate (PBT)	1700A	Celanex Ticona
Rubber AntiOxidant	Flectol TMQ	Flexsys America
Stearic Acid	Emersol-150	Cognis Canada Corp.
Paracin Wax	285	Caschem
Sartomer	SR-525 N,N – m – phenylene dimaleimide	Sartomer Company
Magnesium Oxide	Maglite D	The C.P. Hall Company
Naugard	PHR	Crompton
Antioxidant 2264	2,2'-Methylenebis (6-t-butyl-4-methylphenol)	Aldrich
Zinc oxide	ZOCO-172	Zochem
ZDEDC	Zinc Diethyldithiocarbamate	R.T. Vanderbilt
Peroxide	Trigonox 101 – 45B - pd	Akzo - Nobel

TABLE 1 B: Injection Moulding Conditions of Plastic and Plastic/Rubber Blend Samples

Plastic or Rubber Blend	Zone I (°C)	Zone II (°C)	Zone III (°C)	Nozzle	Mould Temperature (°C)
PP	190	200	220	100%	~40
PP/CIIR 1068	190	200	220	100%	~40
PP/Nitrile	190	200	220	100%	~40
PBT	255	290	230	100%	~60
PBT/Nitrile & Pure PBT	255	290	230	100%	~60
Polyamide 12	180	180	180	100%	~25
Polyamide 12/Nitrile	215	225	180	100%	~25
Polyamide 12/CIIR 1068	180	180	180	100%	~25

TABLE 2: Solvent Selection for Rubber & Plastic Samples

- Rubber Samples: Chlorobutyl 1068, Krynac 3345C
- Plastic Samples: PA, PP, PBT

I. Chlorobutyl rubber

Solvent	% Insolubility
hexane	0
$CHCl_3$	0

- Clorobutyl 1068 is completely soluble in both hexane and $CHCl_3$

II. Nitrile Rubber

Solvent	% Insolubility
MEK	57.1
acetone	58.5
$CHCl_3$	30.4
toluene	24.5
acetic acid	90.2
hexane	96.4

- $CHCl_3$ and Toluene are the best solvents for NBR

lll. Plastics

Sample Description	Solvent	% Insolubility
PBT	MEK	104.3
	toluene	104.8
	$CHCl_3$	105.4
	hexane	98.1
PP	MEK	96.1
	toluene	104.7
	$CHCl_3$	104.2
	hexane	94.1
PA	MEK	105.4
	toluene	103.2
	$CHCl_3$	105.0
	hexane	99.5*

* based on swelling index % insolubility

- Values of % insolubility which exceed 100% are due to a small uptake of the solvent by the plastic. Analysis of the solvent reveals very little dissolution of the plastic sample.

Comments:

1. The purpose for the preceding tests was to establish which solvents dissolved the rubber materials, and which ones the plastic materials.
2. A good solvent for purpose of % solubility and swelling index tests on blends is: rubber sample – low % insolubility (high % solubility), plastic sample – high % insolubility (low % solubility).
3. $CHCl_3$ was chosen as the best solvent for studies of % solubility and swelling index for these blends of rubber and plastic. It is an excellent solvent for Chlorobutyl rubber, and a relatively good solvent for Nitrile rubber. In addition, none of the plastics are soluble in $CHCl_3$.

TABLE 3: Determination of Formulation for NBR cure in NBR/Plastic Blends

Part 1: Sample Preparation of Nitrile Rubber

	Rubber Composition							Moulding Conditions		
ID #	**NBR**	**Sartomer**	**MgO**	**Peroxide**	**Stearic Acid**	**Paracin Wax**	**A/O***	**Cure Temp. °C**	**Cure Time min.**	**Blend Type**
040130-3	100	2.5	-	2.08	1.44	0.55	1.7	210	6	PA/NBR
040414-2	100	2.5	-	2.08	1.44	0.55	1.7	-	-	PA/NBR
040414-1	100	1.7	2.5	-	1.44	0.55	1.7	-	-	PP/NBR
040129-1	100	1.7	-	-	1.44	0.55	1.7	210	6	PBT/NBR
040202-1	100	2.5	2.5	2.08	1.44	0.55	1.7	190	6	no blend

*antioxidant

Part 2: Properties of Pure NBR System

ID #	**Sample Description**	**Ultimate Strength (MPA)**	**Elong. @ Break (%)**	**Modulus (MPA)**	**% Insol. (S.I.) $CHCl_3$**	**Swelling Index ($CHCl_3$)**	**Blend Type**
040130-3	100 NBR with Sartomer & Peroxide	2.7	140	2	96.6	5.99	PA/NBR
040414-2	100 NBR with Sartomer & Peroxide	-	-	-	98.5	5.78	PA/NBR
040414-1	100 NBR with Sartomer & MgO	-	-	-	45.7	34.61	PP/NBR
040129-1	100 NBR with Sartomer	2.3	572	1	79.0	24.43	PBT/NBR
040202-1	100 NBR w/ Sartomer, Peroxide, & MgO	3.2	148	2	96.9	5.61	no blend

Comments:

1. A variety of different formulations were used to cure rubber depending on the plastic used in the blend. These tables indicate the mechanical and solvent properties for nitrile rubber in various formulations.
2. Formulations were based on the results of A.Y. Coran, R.P. Patel, D. Williams, Rubber Chem. Technol. 55, 116, 1982.
3. Swelling index values and mechanical properties in this table provide base values for nitrile blends containing 0% plastic.

TABLE 4: Comparison of Swelling Index of Selected Thermoplastic Resin/ Rubber Blends in Various Solvents

Part 1: Comparison of Swelling Index and Percentage Insolubles for $CHCl_3$ and Toluene in PP/NBR & PBT/NBR Blends

ID#	Sample Description	Swelling Index $CHCl_3$	% Insol. (S.I.) in $CHCl_3$	Swelling Index in Toluene	% Insol. (S.I.) in Toluene
040129-1	100NBR w/ sartomer	24.43	80.0	6.51	88.4
040211-3	15PBT/85NBR	16.16	89.2	2.57	91.8
040209-3	20PBT/80NBR	9.39	86.6	2.23	95.2
040209-2	25PBT/75NBR	7.97	93.1	2.15	95.6
040209-1	30PBT/70NBR	6.29	94.0	1.98	96.0
040206-3	35PBT/65NBR	5.59	94.8	1.86	96.5
040206-2	40PBT/60NBR	4.95	95.3	1.77	96.8
PBT	100PBT	1.15	102.4	1.01	100
	average SD (%):	*4.21*	*1.33*	*1.56*	*0.96*
040414-1	100NBR w/ sart. & MgO	36.61	45.7	9.90	76.3
040206-1	15PP/85NBR	10.59	87.1	3.05	89.1
040205-3	20PP/80NBR	9.83	87.7	3.02	89.4
040205-2	25PP/75NBR	8.75	87.4	2.83	90.1
040205-1	30PP/70NBR	7.75	87.3	2.62	90.8
040204-3	35PP/65NBR	7.14	88.1	2.45	92.2
040204-1	40PP/60NBR	6.23	87.3	2.29	91.8
PP	100PP	1.26	99.7	1.15	99.3
	average SD (%):	*5.36*	*7.28*	*1.16*	*0.22*

- $CHCl_3$ causes substantial swelling in blends of PBT/NBR and PP/NBR. Toluene swells similar blends to a lesser extent.

Part 2: Comparison of Swelling Index and Percentage Insolubles in Hexane versus $CHCl_3$ for PA/CIIR (ZDEDC-cured)

ID#	Sample Description[1]	Swelling Index Hexane	% Insol. (S.I.) in Hexane	Swelling Index in $CHCl_3$	% Insol. (S.I.) in $CHCl_3$
010829-2	100CIIR[2]	2.93	99.9	6.51	99.6
040311-1	15PA/85CIIR	3.59	68.4	8.33	67.0
040310-4	20PA/80CIIR	2.55	81.3	5.60	78.9
040310-3	25PA/75CIIR	1.88	92.0	3.90	90.8
040310-2	30PA/70CIIR	1.60	95.6	3.18	94.3
040310-1	35PA/75CIIR	1.45	97.2	2.66	97.2
040309-2	40PA/60CIIR	1.34	99.1	2.35	98.3
N12	100PA[3]	1.00	99.5	1.08	104.8
	average SD (%)	*0.73*	*0.2*	*1.32*	*0.41*

Notes:
1. All blends with CIIR cured with 2.2 phr ZDEDC based on rubber, and injection moulded.
2. Sample cured in press.
3. Sample pressed, 1.5 mm thickness.

Comments:
1. Both hexane and $CHCl_3$ swell PA/CIIR blends. In comparison to hexane, the higher swelling index for $CHCl_3$ in PA/CIIR blends is primarily due to higher density. For PP/NBR and PBT/NBR blends, the higher $CHCl_3$ values over toluene are attributed to both density and higher compatibility (as measured by solubility parameter).
2. Overall, for comparison between different blend systems, $CHCl_3$ is the best solvent, as all blends become measurably swollen upon exposure.

TABLE 5: Mechanical & Solution Properties of Polyamide / Chlorobutyl Blends[1, 2]

Part 1: Properties

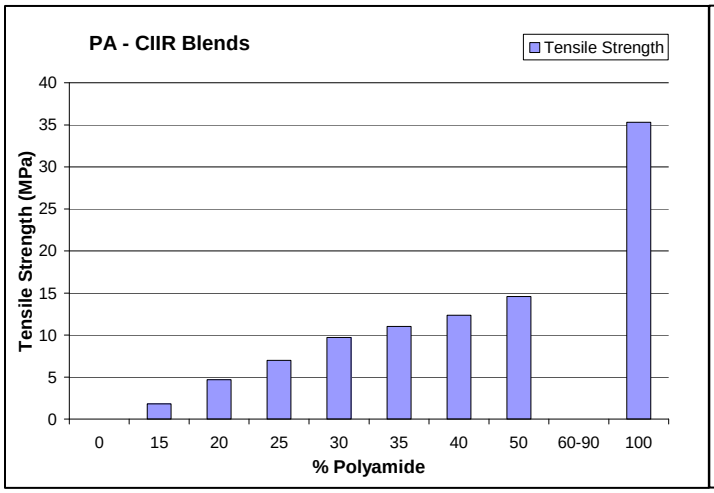
		Mechanical Properties				Solution Properties			
Sample No.	Blend Ratio (PA/CIIR)	Ultimate Strength (Mpa)[3]	Elong. @ Break (%)[4, 5]	Modulus (Mpa)[6]	Shore D Hard-ness[7]	Swelling Index (Hexane)[8]	Swelling Index ($CHCl_3$)[9]	% Insol. (S.I.) Hexane[10]	% Insol. (S.I.) $CHCl_3$[11]
010829-2	0/100					2.93	6.51	99.9	99.6
040311-1	15/85	1.8	252	3.7	12	3.59	8.33	68.4	67.0
040310-4	20/80	4.7	168	16.3	22	2.55	5.60	81.3	78.9
040310-3	25/75	7.0	165	35.8	30	1.88	3.90	92.0	90.8
040310-2	30/70	9.7	196	74.4	37	1.59	3.18	95.6	94.3
040310-1	35/65	11.0	210	77.5	46	1.45	2.66	97.2	97.2
040309-2	40/60	12.3	195	91.7	49	1.34	2.34	99.1	98.3
040601-1	50/50	14.6	94				2.06		97.2
040615-1	100/0	35.3	110			1.00	1.08	99.5	104.8

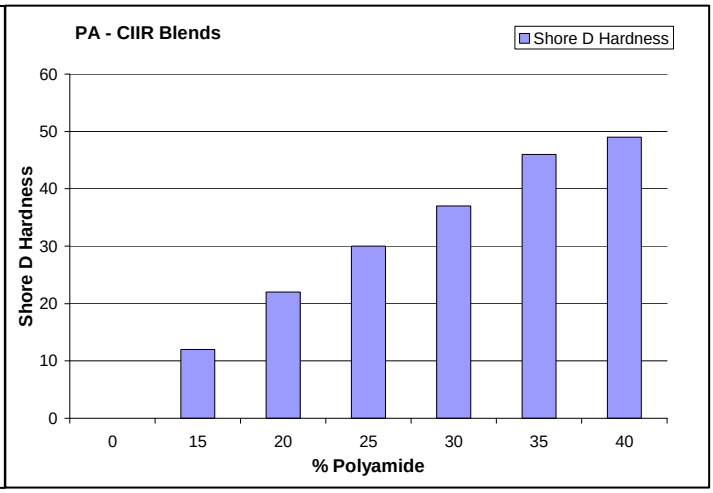
1. Formulation: Nylon 12 (L16), Chlorobutyl 1068, ZnO 6.70 phr, Stearic acid 1.44 phr, Paracin wax 0.55 phr, ZDEDC 2.2 phr. 2. Samples injection moulded, except for 0/100 which was compression molded. 3. Average SD of 4%. 4. Elongation at Break values based on bench marks. 5. Average SD of 5%. 6. Average SD of 13%. 7. Average SD of 4%. 8. Average SD of 0.7%. 9. Average SD of 1.3%. 10. Average SD of 0.2%. 11. Average SD of 0.4%.

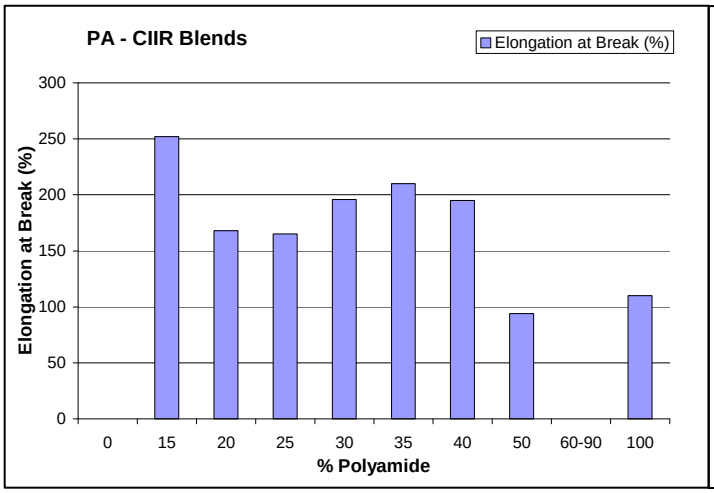
Part 2: Values of Standard Deviation for Blends Listed Above

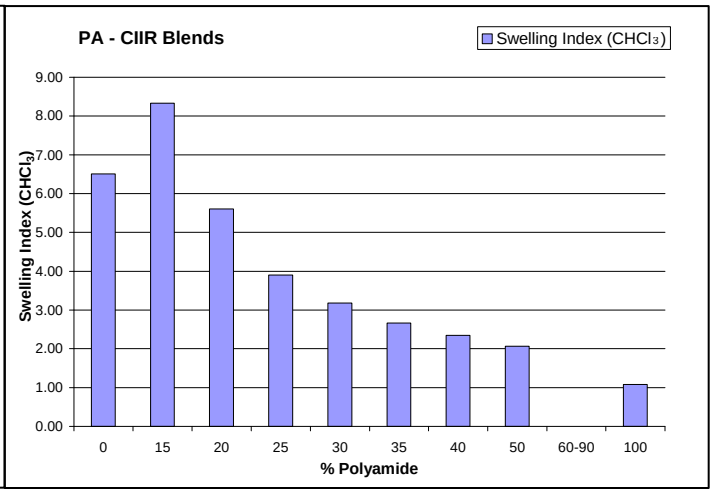
		Mechanical Properties				Solution Properties			
Sample No.	Blend Ratio (PA/CIIR)	SD Ultimate Strength	SD Elong.	SD Modulus	SD Shore D	SD S. I. (Hexane)	SD S.I. ($CHCl_3$)	SD % Insol. (Hexane)	SD % Insol. (CHCl3)
010829-2	0/100								
040311-1	15/85	0.15	8	0.15	1	0.05	0.14	0.40	0.34
040310-4	20/80	0.29	5	2.5	1	0.05	0.05	0.20	0.53
040310-3	25/75	0.07	4	2.0	1	0.05	0.18	0.37	1.25
040310-2	30/70	0.32	5	24.7	1	0.01	0.05	0.19	0.95
040310-1	35/65	0.49	5	9.1	1	0.00	0.05	0.23	0.30
040309-2	40/60	0.11	5	7.7	1	0.00	0.01	0.35	0.07
040601-1	50/50	0.26	5				0.02		0.57
040615-1	100/0	5.90	60						
Average SD (%)		4.00	5	13	7	0.7	1.3	0.2	0.4

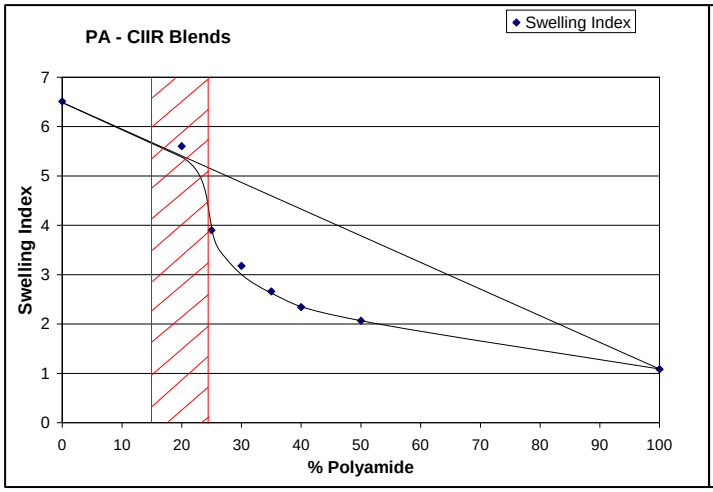
Note: This table lists standard deviation values for the tests conducted on the blends listed in Part 1. Average values (in percent) are also listed. Only the values of average % standard deviation are listed in Tables 6 to 9.

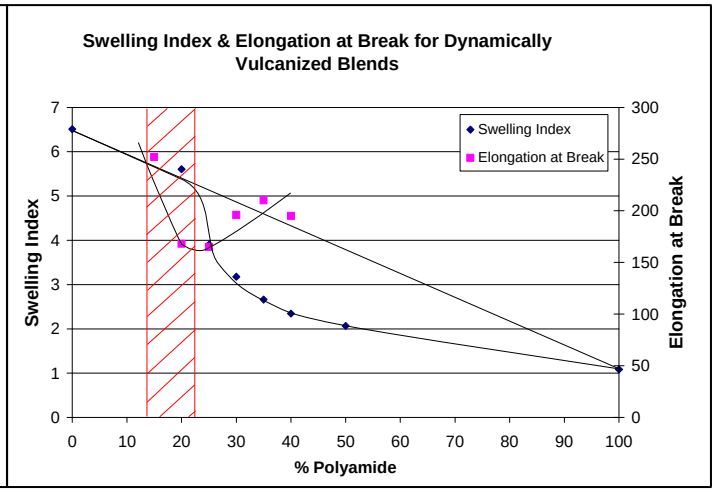












Comments:

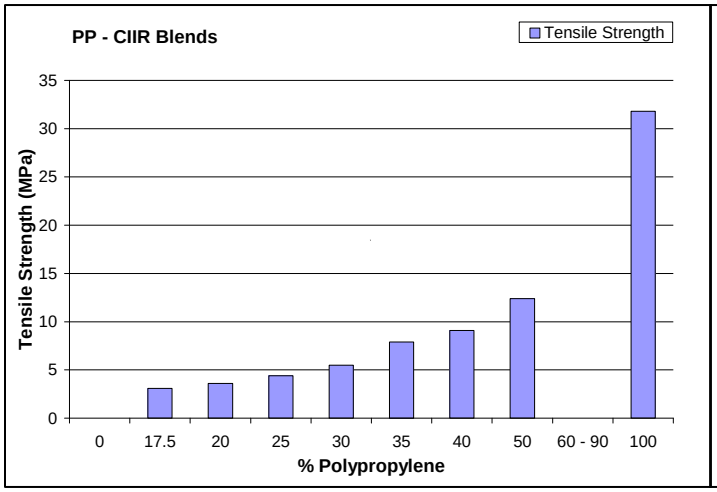
1. This data was obtained from project W7702 R905/001/EDM.
2. The hatched area indicates the approximate composition where phase inversion occurs.
3. Past phase inversion the S.I. values are consistently below theoretical line (physical mixture). Here also the continuous thermoplastic phase prevents solvent expansion of cured rubber phase.
4. A minimum value in elongation at break is reached just past phase inversion. (Values at high % polyamide are not included in this plot.)

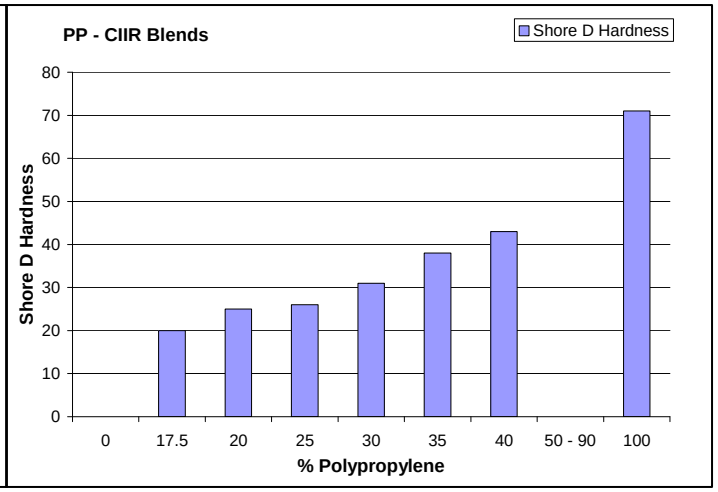
TABLE 6: Mechanical & Solution Properties of Polypropylene / Chlorobutyl Blends[1, 2]

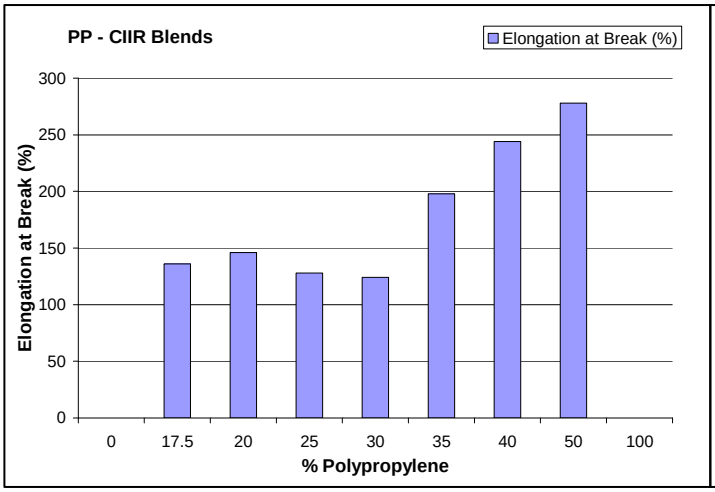
		Mechanical Properties				**Solution Properties**			
Sample No.	Blend Ratio (PP/CIIR)	Ultimate Strength (Mpa)[3]	Elong. @ Break (%)[4, 5]	Modulus (Mpa)[6]	Shore D Hard-ness[7]	Swelling Index (Hexane)[8]	Swelling Index ($CHCl_3$)[9]	% Insol. (S.I.) Hexane[10]	% Insol. (S.I.) $CHCl_3$[11]
010829-2	0/100					2.93	6.51	99.9	99.6
040312-5	17.5/82.5	3.1	136	13	20	2.62	5.16	87.6	86.8
040312-4	20/80	3.6	146	19	25	2.45	5.05	89.2	87.8
040312-3	25/75	4.4	128	30	26	2.30	4.48	89.3	88.0
040312-2	30/70	5.5	124	41	31	2.09	3.84	91.7	91.6
040312-1	35/65	7.9	198	67	38	1.87	3.43	94.3	93.0
040311-2	40/60	9.1	244	79	43	1.77	2.97	94.5	94.2
040601-2	50/50	12.4	278			n/a	2.47		95.7
40126	100/0	31.8		138	71	n/a	1.26	94.1*	99.7

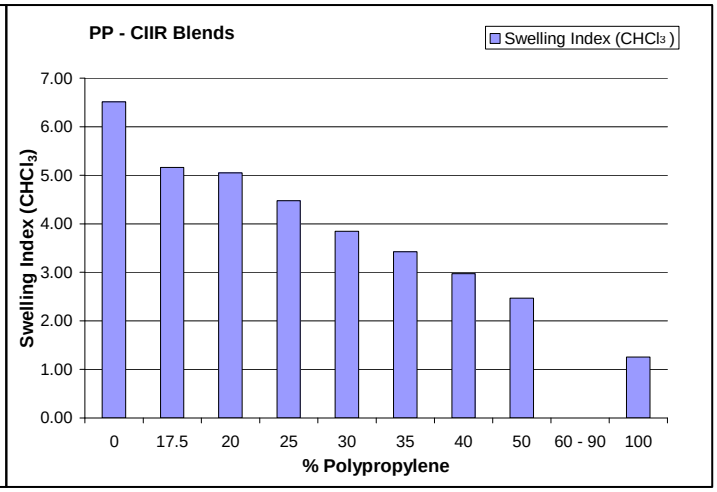
* based on % Insol. Test, not S.I.

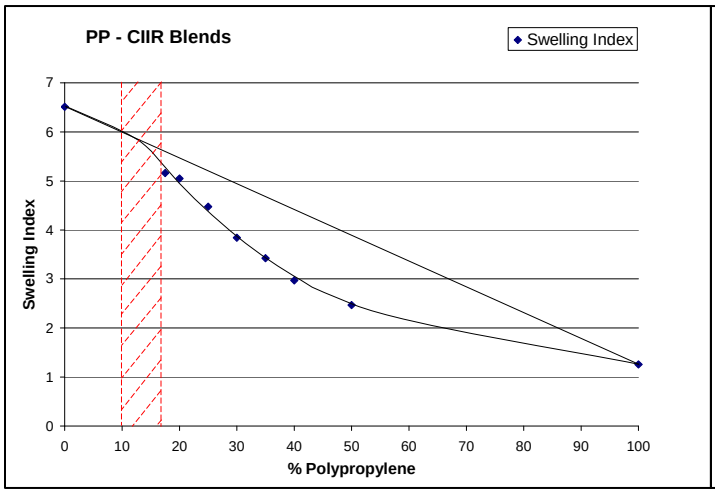
1. Formulation: Polypropylene, Chlorobutyl 1068, ZnO 6.70 phr, Stearic acid 1.44 phr, Paracin wax 0.55 phr, ZDEDC 2.2 phr. 2. injection molded, except for 0/100 which was compression molded. [3]. Average SD of 4%. [4]. Elongation at Break values based on bench marks. [5]. Average SD of 5%. [6]. Average SD of 13%. [7]. Average SD of 4%. [8]. Average SD of 0.7%. [9]. Average SD of 1.5%. 10. AverageSD of 0.4%. 11. Average SD of 0.4%.

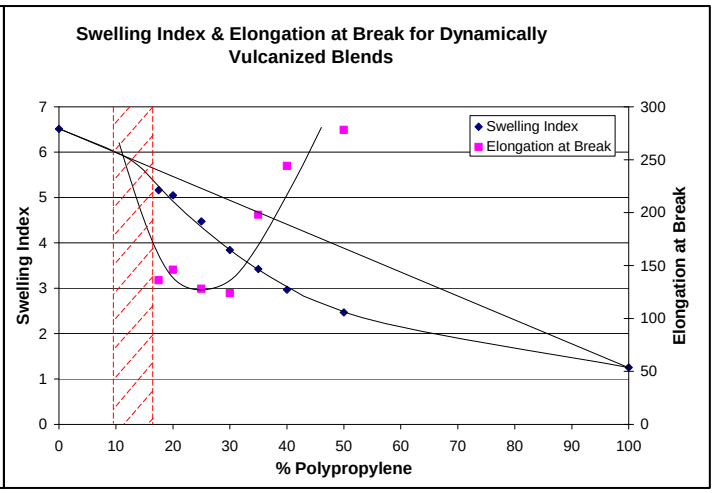












Comments:

1. The hatched area indicates the approximate composition where phase inversion is thought to occur.
2. Past phase inversion the S.I. values are consistently below theoretical line (physical mixture). Here the continuous thermoplastic phase prevents solvent expansion of cured rubber phase.
3. A perceived minimum value in elongation at break is reached just past phase inversion.

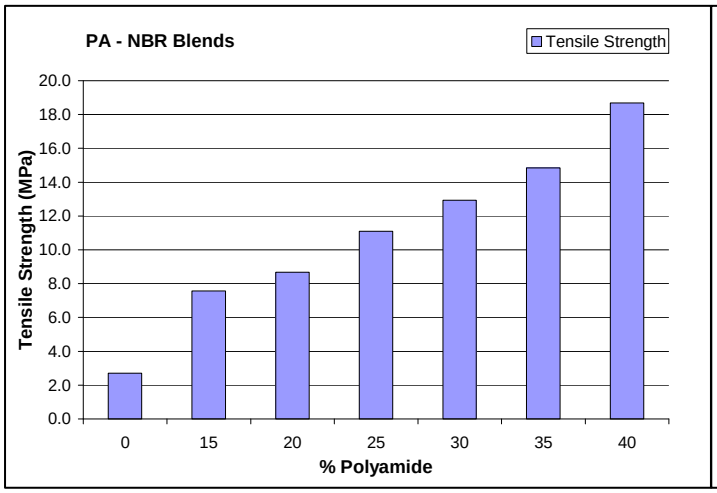
TABLE 7: Mechanical & Solution Properties of Polyamide / Nitrile Rubber Blends[1, 2]

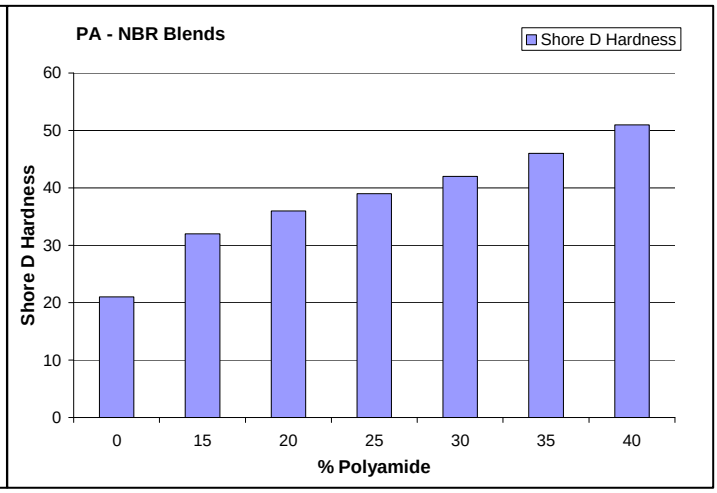
		Mechanical Properties				**Solution Properties**	
Sample No.	Blend Ratio (PA/NBR)	Ultimate Strength (Mpa)[3]	Elong. @ Break (%)[4, 5]	Modulus (Mpa)[6]	Shore D Hard-ness[7]	Swelling Index ($CHCl_3$)[8]	% Insol. (S.I.) $CHCl_3$[9]
040130-3	0/100	2.7	140	1.8	21	5.99	96.6
040211-2	15/85	7.6	86	25.2	32	5.26	95.3
040211-1	20/80	8.7	128	31.5	36	5.57	94.7
040210-3	25/75	11.1	148	33.2	39	4.99	95.3
040210-2	30/70	12.9	164	47.5	42	4.67	94.8
040210-1	35/65	14.8	176	61.1	46	4.14	93.9
040202-2	40/60	18.7	192	76.8	51	4.31	97.0
N12	100/0					1.08	104.8

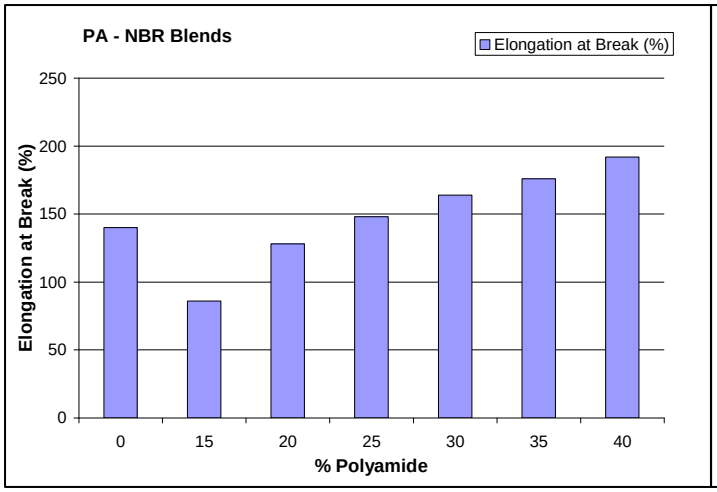
1. Formlation: Nylon 12 (L16), Nitrile, [Sartomer 2.50 phr, peroxide 2.1 phr, Antioxidant TMQ 1.66 phr, all based on rubber], [Stearic acid 1.44 phr, Paracin Wax 0.55 phr, based on total polymer]. 2. injection molded, except for 0/100 which was compression molded. 3. Average SD of 4%. 4. Elongation at Break values based on bench marks. 5. Average SD of 5%. 6. Average SD of 13%. 7. Average SD of 4%. 8. Average SD of 2.4%. 9. Average SD of 1.9%.

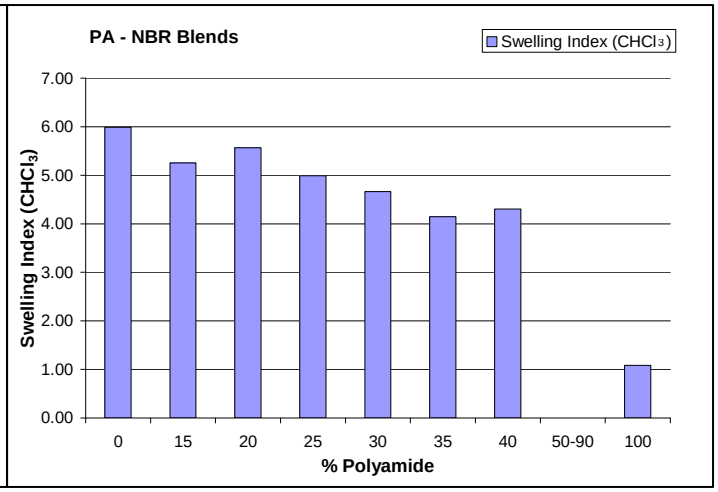
Comments:

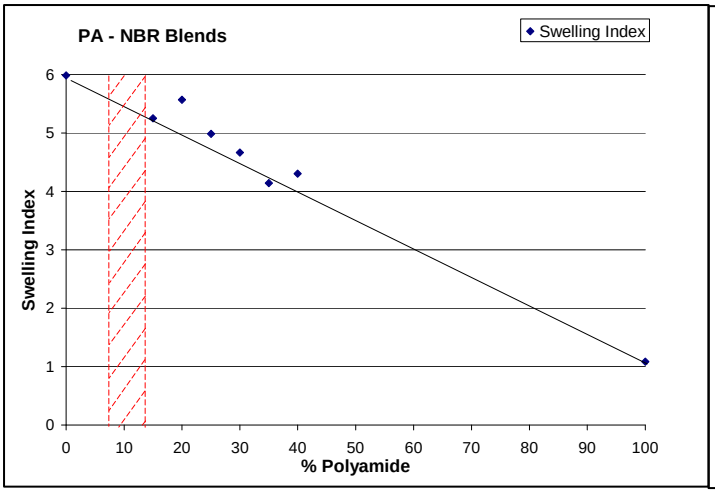
1. PA/NBR blends continued to crosslink after blending. After one month, samples in this series had lower swelling index values by 0.5 – 1 points.

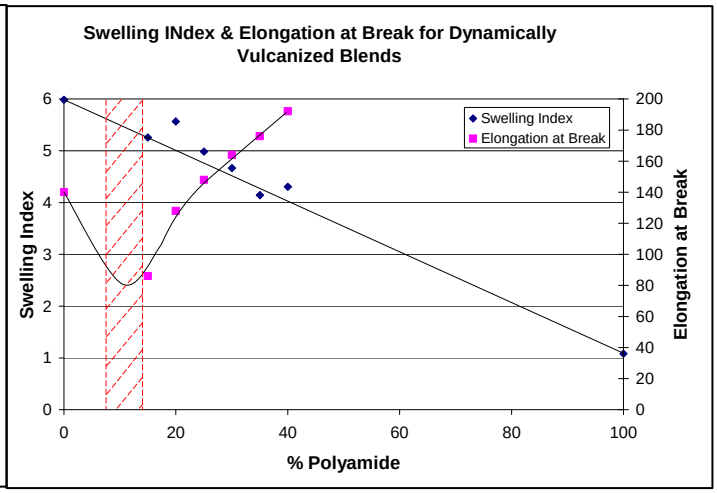












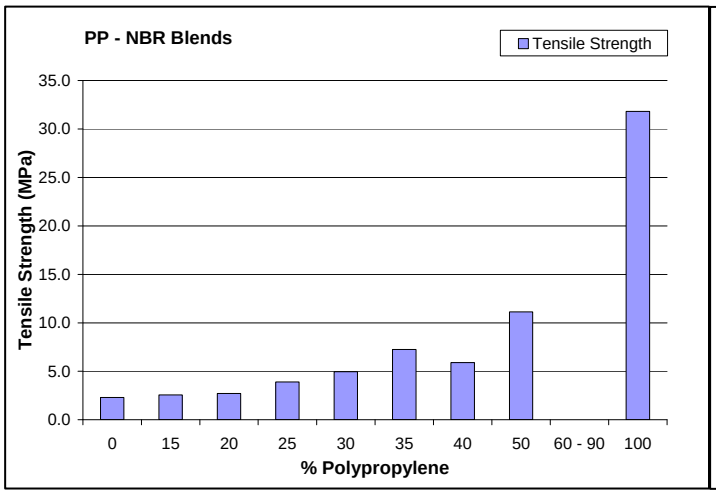
Comments:

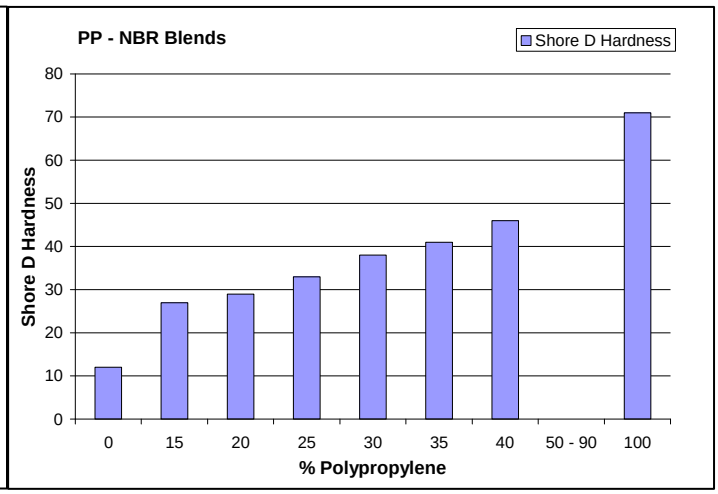
1. The hatched area indicates the approximate composition where phase inversion is thought to occur.
2. Because of the compatibility between PA and NBR the swelling index values never go below the theoretical line (physical mixture).
3. A minimum value in elongation at break is reached just past phase inversion.

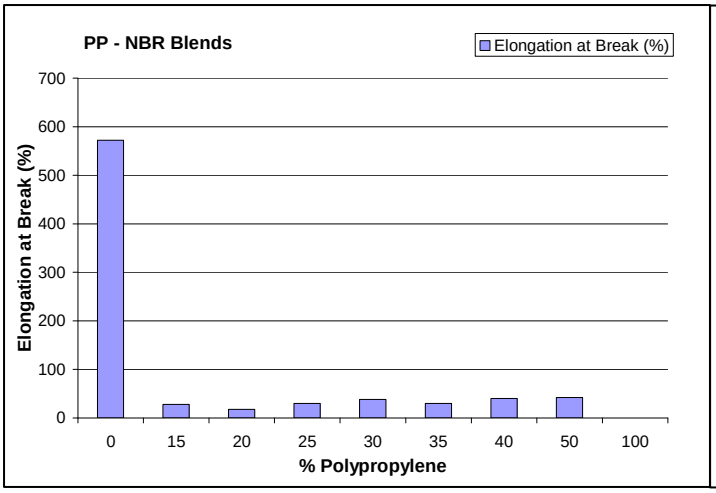
TABLE 8: Mechanical & Solution Properties of Polypropylene / Nitrile Rubber Blends[1, 2]

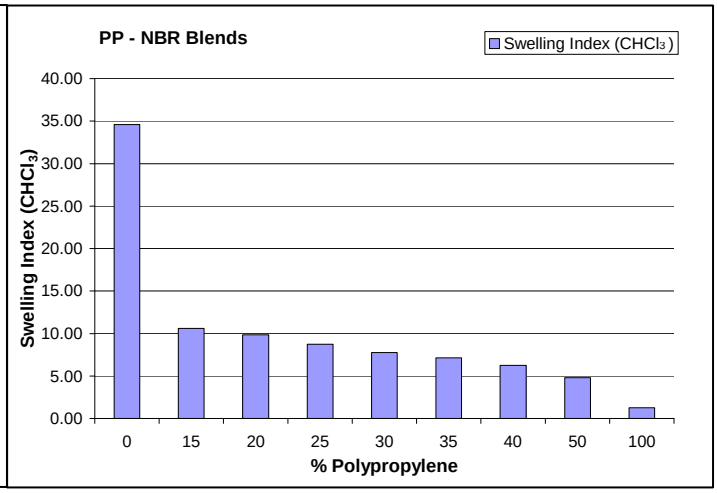
		Mechanical Properties				**Solution Properties**	
Sample No.	Blend Ratio (PP/NBR)	Ultimate Strength (Mpa)[3]	Elong. @ Break (%)[4, 5]	Modulus (Mpa)[6]	Shore D Hard-ness[7]	Swelling Index ($CHCl_3$)[8]	% Insol. (S.I.) $CHCl_3$ [9]
040414-1	0/100	2.3	572	0.7	12	34.61	45.7
040206-1	15/85	2.6	28	16.9	27	10.59	87.1
040205-3	20/80	2.7	18	22.6	29	9.83	87.7
040205-2	25/75	3.9	30	33.6	33	8.75	87.4
040205-1	30/70	4.9	38	43.5	38	7.75	87.3
040204-3	35/65	7.2	30	78.1	41	7.14	88.1
040204-1	40/60	5.9	40	54.9	46	6.23	87.3
040601-3	50/50	11.1	42			4.80	91.0
40126	100/0	31.8		138.0	71	1.26	99.7

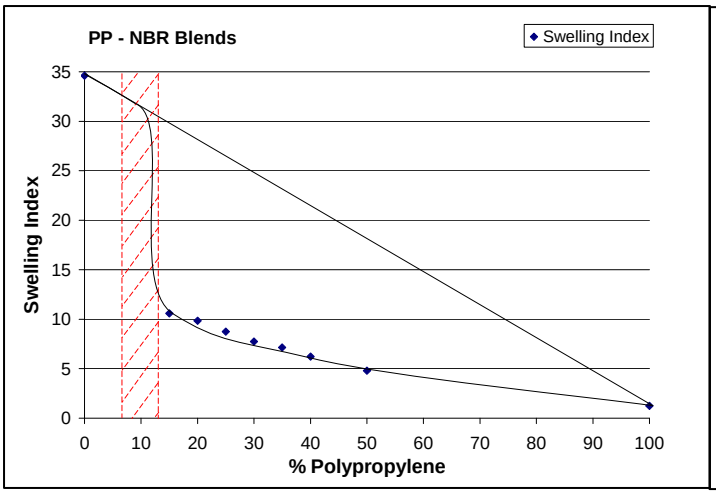
1. Formlation: Polypropylene, Nitrile, [Sartomer 2.50 phr, Antioxidant 1.67 phr, Naugard PHR 0.83 phr, based on rubber], [Stearic acid 1.44 phr, Paracin Wax 0.55, MgO 2.50 phr, based on total polymer]. 2. injection molded, except for 0/100 which was compression molded. 3. Average SD of 4%. 4. Elongation at Break values based on bench marks. 5. Average SD of 5%. 6. Average SD of 13%. 7. Average SD of 4%. 8. Average SD of 5.4%. 9. Average SD of 7.3%.

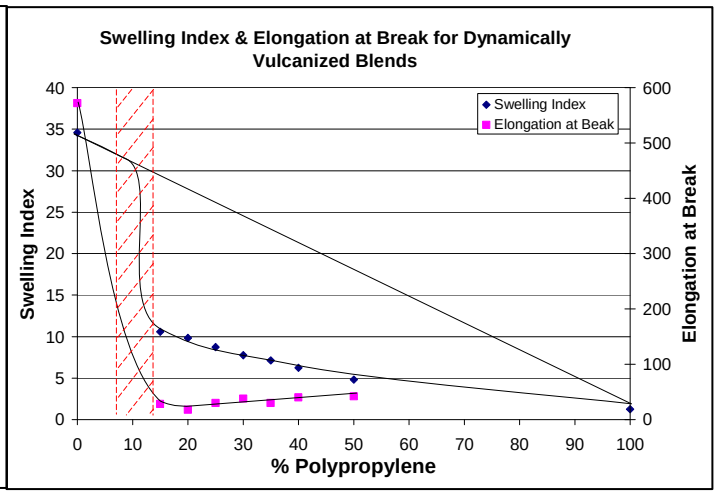












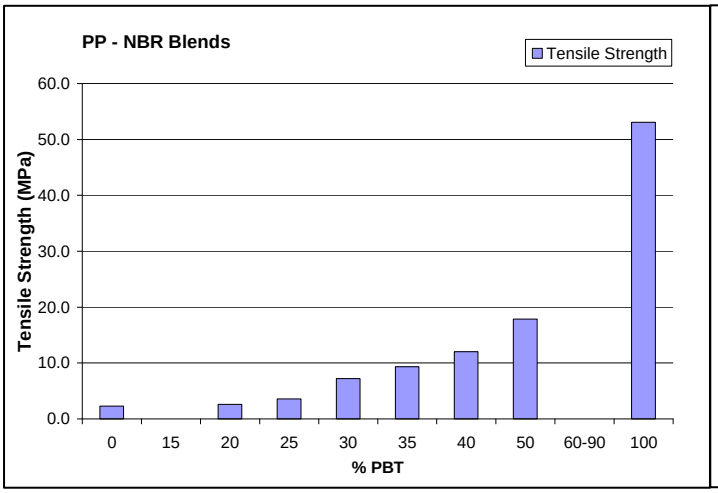
Comments:

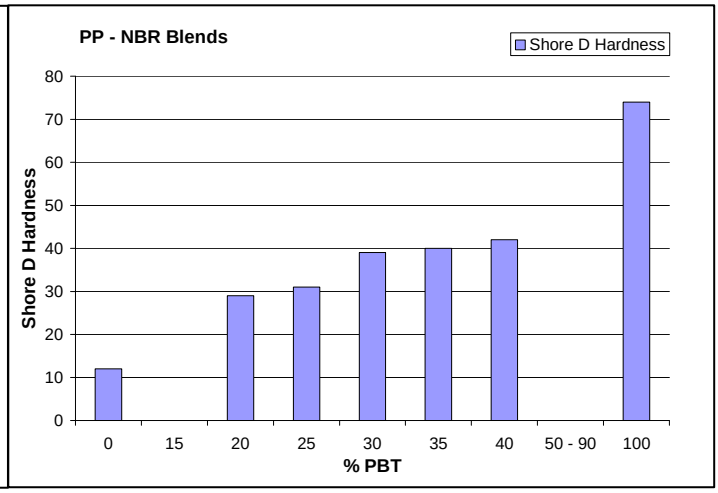
1. The hatched area indicates the approximate composition where phase inversion is thought to occur.
2. Past phase inversion the S.I. values are dramatically below the theoretical line (physical mixture). Here the continuous thermoplastic phase prevents solvent expansion of cured rubber phase.
3. The elongation at break is at its minimum value at a point just past phase inversion.

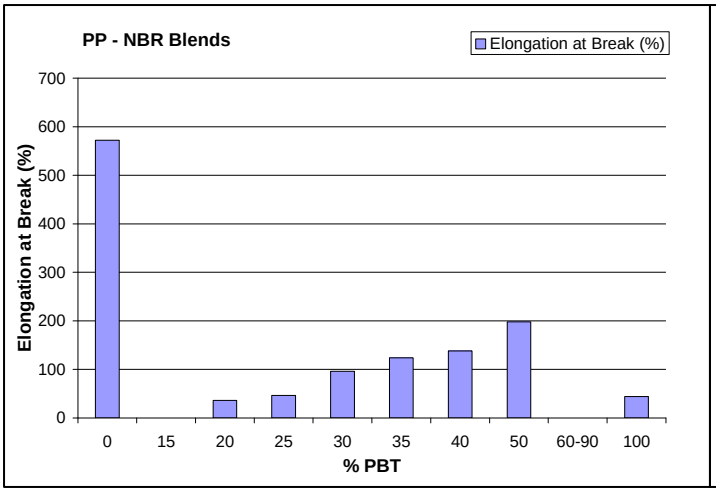
TABLE 9: Mechanical Properties & Hardness of Polybutyl Terephthlate / Nitrile Rubber Blends[1, 2]

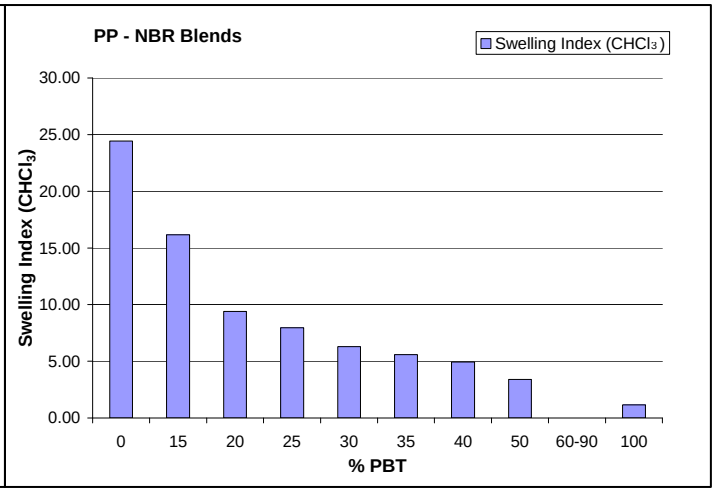
		Mechanical Properties				**Solution Properties**	
Sample No.	Blend Ratio (PBT/NBR)	Ultimate Strength (Mpa)[3]	Elong. @ Break (%)[4, 5]	Modulus (Mpa)[6]	Shore D Hard-ness[7]	Swelling Index ($CHCl_3$)[8]	% Insol. (S.I.) $CHCl_3$ [9]
040129-1	0/100	2.3	572	0.7	12	24.43	79.0
040211-3	15/85					16.16	89.2
040209-3	20/80	2.6	36	13.2	29	9.39	86.6
040209-2	25/75	3.6	46	17.4	31	7.97	93.1
040209-1	30/70	7.2	96	26.7	39	6.29	94.0
040206-3	35/65	9.4	124	36.0	40	5.59	94.8
040206-2	40/60	12.1	138	48.1	42	4.95	95.3
040601-4	50/50	17.9	198			3.39	97.6
Natural PBT	100/0	53.1	44	458.4	74	1.15	102.4

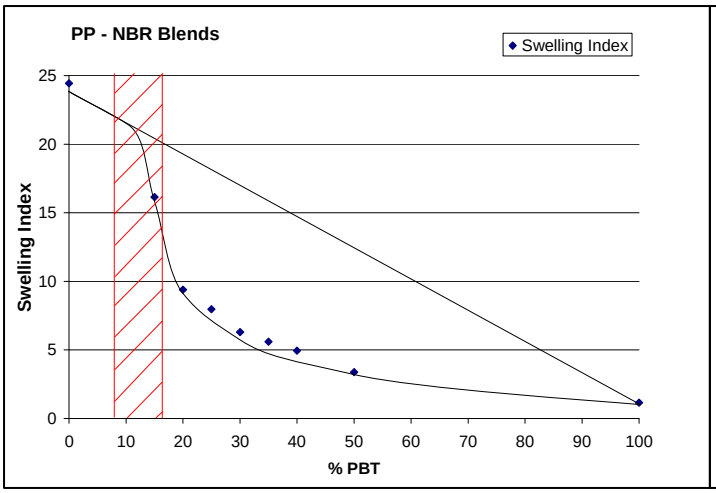
1. Formulation: PBT, Nitrile, [Stearic acid 1.44 phr, Paracin wax 0.55 phr, based on total polymer], [Antioxidant TMQ 1.56 phr, Sartomer 1.56 phr, based on rubber]. 2. injection molded, except for 0/100 which was compression molded. 3. Average SD of 4%. 4. Elongation at Break values based on bench marks. 5. Average SD of 5%. 6. Average SD of 13%. 7. Average SD of 4%. 8. Average SD of 4.2%. 9. Average SD of 1.3%.

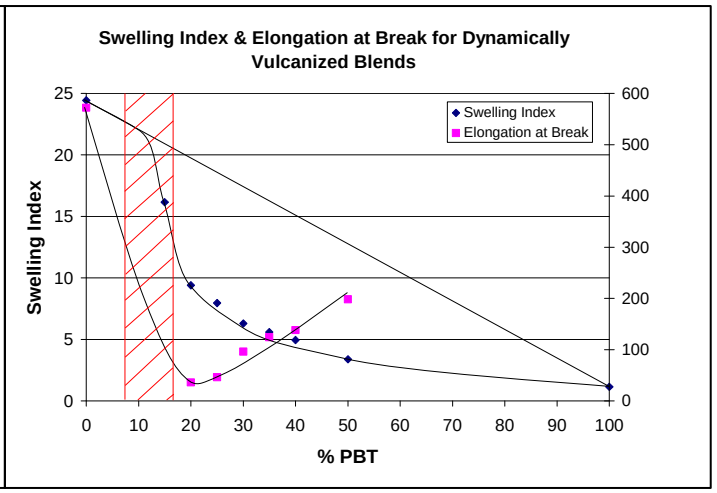












Comments:

1. The hatched area indicates the composition where phase inversion occurs.
2. All values of S.I. are below the theoretical line (physical mixture), including where phase inversion is occurring. Past phase inversion the continuous thermoplastic phase prevents solvent expansion of the cured rubber phase.
3. The elongation at break is at its minimum value at a point just past phase inversion.

TABLE 10: HD Penetration / Re-emission Data for Rubber / Plastic Blends

Test Conditions:
Closed Cell

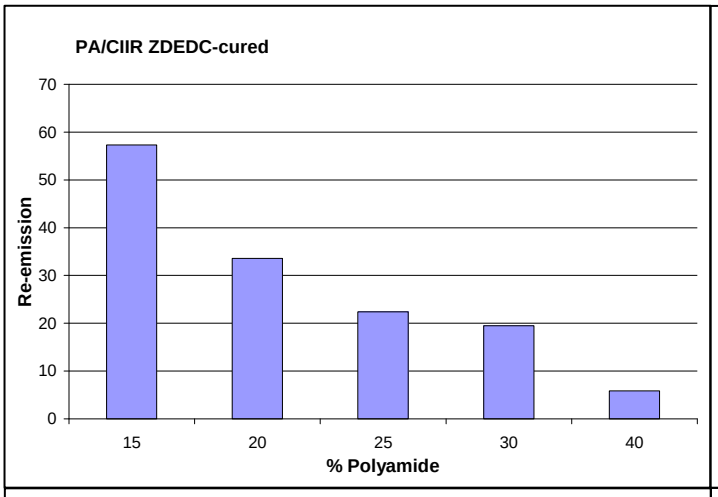
Temperature:	30C
Collection Rate:	100 ml/min
Collection Time:	24 Hrs
Absorbant solvent:	5ml diethyl succinate
Challenge:	5 X 1 ul drop

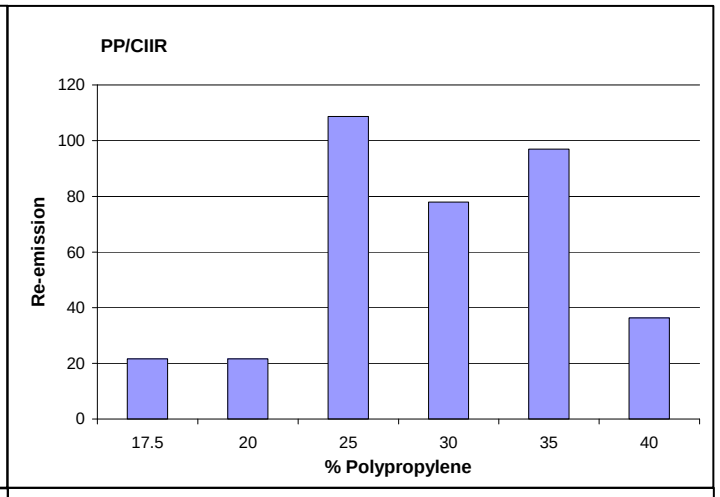
Sample No.	Blends Ratio	Pene-tration	Re-emission	Sample No.	Blends Ratio	Pene-tration	Re-emission
PA/CIIR ZDEDC-cured				**PA/CIIR sulfur-cured**			
030320-2	15/85		57.3	030129-2	15/85		37.2
030320-1	20/80		33.6	030129-1	20/80		28.3
030318-1	25/75		22.4	030128-2	25/75		17.9
030314-1	30/60		19.5	030128-1	30/70		9.7
010523-1	40/60		5.9	010717-1	40/60		0
nat PA	100/0			nat PA	100/0		

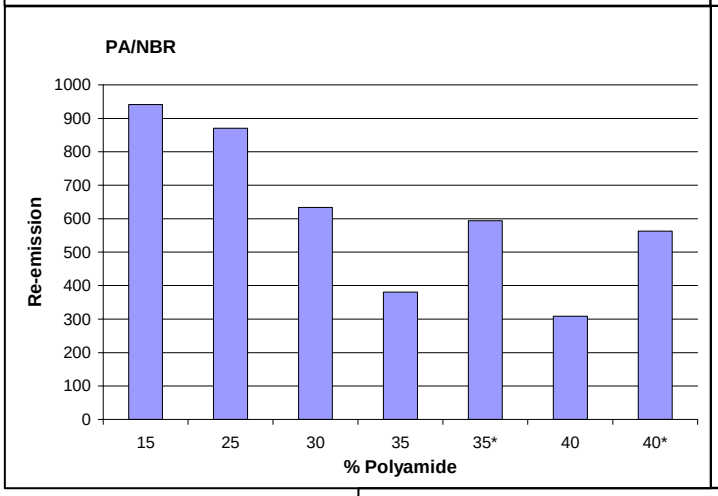
PP/CIIR				**PA/NBR**			
040130-1	17.5/82.5	0	21.6	040211-2	15/85	0	941
040126-4	20/80	0	21.6	040210-3	25/75	0	870
040126-3	25/75	0	108.7	040210-2	30/70	0	634
040126-2	30/70	0	77.9	040210-1	35/65	0	381
040129-3	35/65	0	97	040130-2	35/65*	0	594
040126-1	40/60	8.2	36.3	040202-2	40/60		309
nat PP	100/0	1.5	0	040129-2	40/60*	0	563
				nat PA	100/0	0	

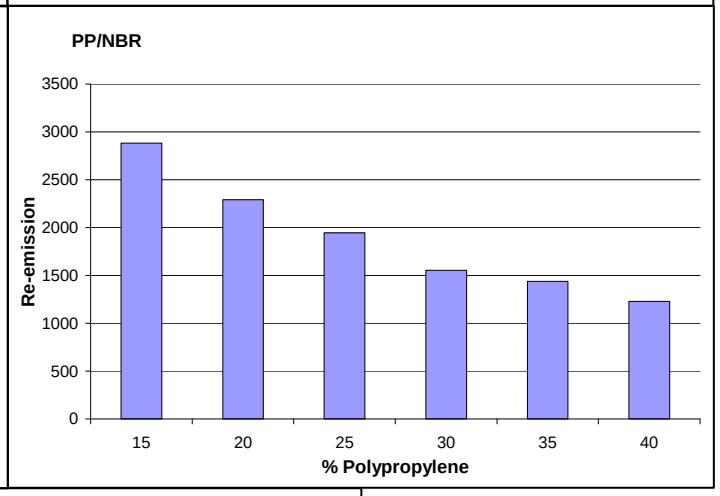
* no Peroxide

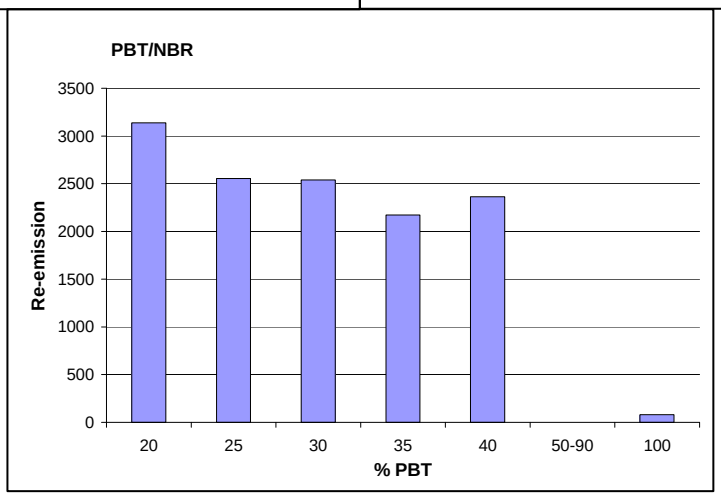
PP/NBR				**PBT/NBR**			
040206-1	15/85	0	2883	040209-3	20/80	0	3138
040205-3	20/80	0	2291	040209-2	25/75	0	2554
040205-2	25/75	0	1945	040209-1	30/70	0	2538
040205-1	30/70	0	1555	040206-3	35/65	0	2173
040204-3	35/65	0	1438	040206-2	40/60	0	2363
040204-1	40/60	0	1230	nat PBT	100/0	7.7	80
nat PP	100/0	1.5	0				











Comments:

1. NBR – containing blends are generally less successful at reducing re-emission values than CIIR – containing blends.
2. In most cases the re-emission values become less as the % thermoplastic is increased.
3. A comparison of the five blends yields the following order for % re-emission (from best to worst):
   PA/CIIR > PP/CIIR > PA/NBR > PP/NBR > PBT/NBR

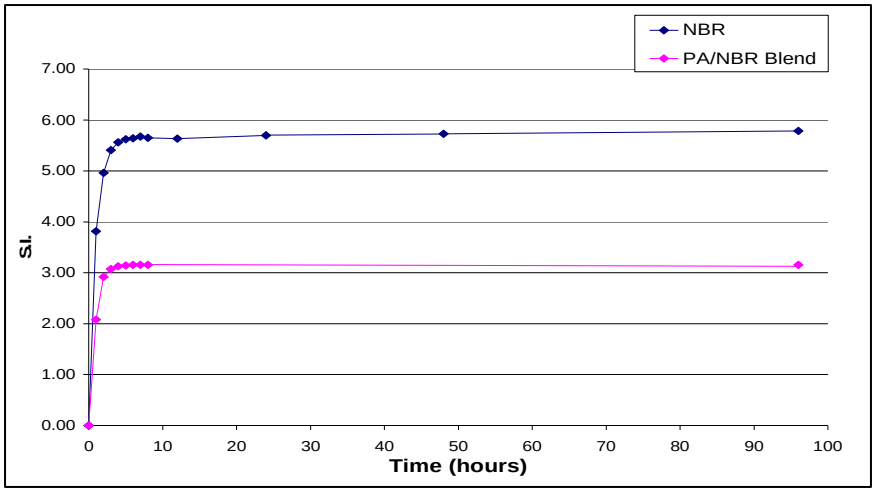
Table 11: Kinetics of Swelling in Chloroform Solvent for Nitrile Rubber Samples & 60:40 Nitrile Rubber / Plastic Blends

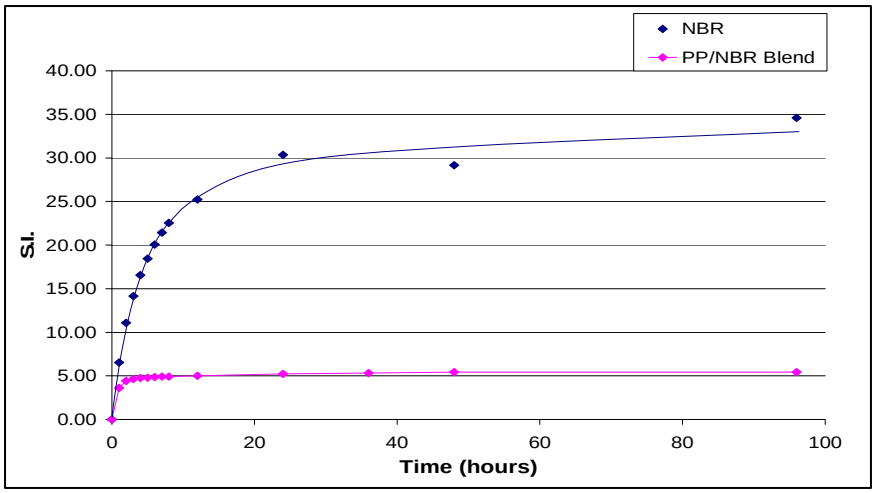
		Average Swelling Index – Rubber Samples[1]			Average Swelling Index – Rubber/ Plastic Samples[2]		
	Type of Blend	PA/NBR[3]	PP/NBR[3]	PBT/NBR[3]	PA/NBR	PP/NBR	PBT/NBR
	Sample No.	040414-2[4]	040414-1[5]	040129-1[6]	040202-2[7]	040204-1[8]	040206-2[9]
Time (hours)	0	0.00	0.00	0.00	0.00	0.00	0.00
	1	3.81	6.53	7.44	2.08	3.63	3.61
	2	4.96	11.08	10.96	2.92	4.43	4.08
	3	5.41	14.15	13.24	3.07	4.64	4.15
	4	5.56	16.56	14.94	3.13	4.75	4.18
	5	5.62	18.43	16.31	3.14	4.79	4.18
	6	5.64	20.05	17.43	3.16	4.86	4.17
	7	5.67	21.45	18.21	3.16	4.91	4.21
	8	5.65	22.54	19.11	3.16	4.92	4.21
	12	5.63	25.25	20.88		5.01	4.23
	24	5.70	30.35	22.24		5.23	4.25
	36					5.33	4.26
	48	5.73	29.16	22.84		5.44	4.26
	96	5.78	34.61	24.43	3.15	5.44	4.25

1. All samples 100 phr NBR with appropriate curing system used in blend.
2. All samples contain 40 phr plastic in a Rubber/Plastic blend.
3. Swelling Index numbers are for rubber samples with formulation corresponding to the blend type indicated.
4. NBR 100 phr, Satomer 2.5 phr, Peroxide 2.1 phr, Antioxidant TMQ 1.66 phr, Steric Acid 1.44 phr, Paracin wax 0.55 phr.
5. NBR 100 phr, Sartomer 2.5 phr, MgO 2.5 phr, Antioxidant 1.67 phr, Naugard PHR 0.83 phr, Stearic Acid 1..44 phr, Paracin wax 0.55 phr.
6. NBR 100 phr, Sartomer 1.56 phr, Antioxidant TMQ 1.56 phr, Stearic acid 1.44 phr, Paracin Wax 0.55 phr.
7. PA 40 phr, NBR 60 phr, Sartomer 1.50 phr, peroxide 1.25 phr, Antioxidant TMQ 1.0 phr, Stearic acid 1.44 phr, Paracin wax 0.55 phr.
8. PP 40 phr, NBR 60 phr, Sartomer 1.50 phr, MgO 2.5 phr, Antioxidant TMQ 1.0 phr, Naugard PHR 0.5 phr, Stearic acid 1.44 phr, Paracin wax 0.55 phr.
9. PBT 40 phr, NBR 60 phr, Sartomer 1.0 phr, Antioxidant TMQ 1.0 phr, Stearic acid 1.44 phr, Paracin wax 0.55 phr.

Comments:

1. NBR rubber samples cured in a variety of different ways rapidly swell under exposure to solvents. Within one day's exposure to solvent, the S.I. value approaches its equilibrium value.
2. NBR blends with PA, PP, or PBT reach equilibrium swelling within 10 hours of exposure to solvent.


NBR
PA/NBR Blend
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
S.I.
0
10
20
30
40
50
60
70
80
90
100
Time (hours)


NBR
PP/NBR Blend
40.00
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
S.I.
0
20
40
60
80
100
Time (hours)

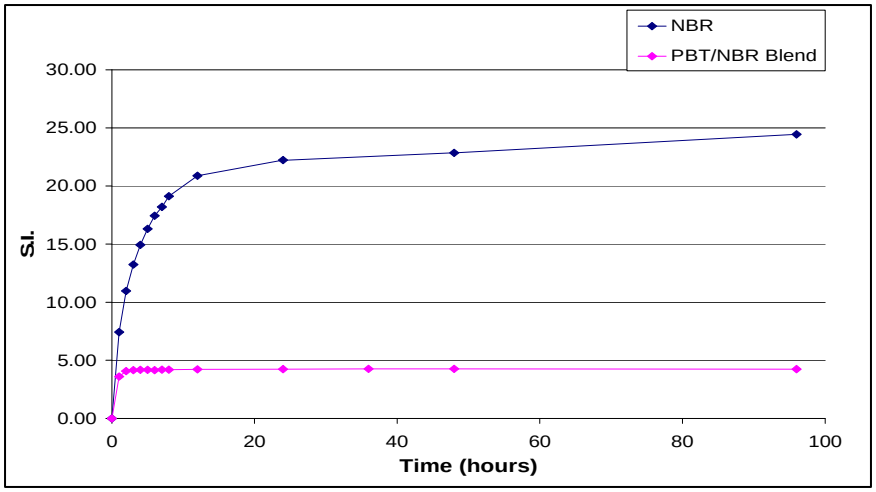

NBR
PBT/NBR Blend
30.00
25.00
20.00
15.00
10.00
5.00
0.00
S.I.
0
20
40
60
80
100
Time (hours)

TABLE 12: DSC Results – Thermoplastic Phase

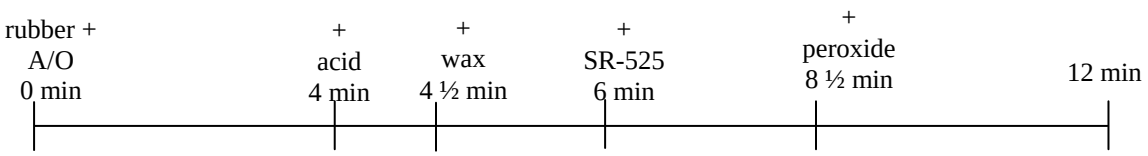
	Tm (^{o}C) (specimens not blended)	**ΔH_f (J/g plastic) (specimens not blended)**	**Tm (^{o}C) (blended and injection)**	**ΔH_f (J/g plastic) (blended and injection molded)**
PA	178.7	60.6		
PA/CIIR	178.8	57.0	175.6	58.5
PA/NBR	178.9	62.5	176.5	58.5
PP	163.3	80.9		
PP/CIIR	160.3	76.6	161.6	83.1
PP/NBR	160.2	78.5	161.5	80.6
PBT	223.1	38.1		
PBT/NBR	222.6	46.0	224.4	113.9

Comments:

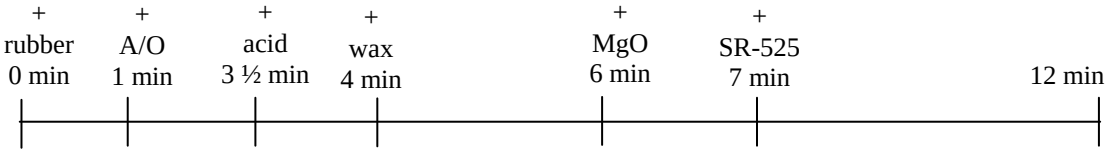
1. The thermoplastic phase exhibits a discrete melting temperature in all blends. This is an indication of phase separation.
2. For PA and PP containing blends the value of Tm is reduced in comparison to the pure resin, indicating that the thermoplastic phase is affected during blending.
3. The high ΔH_f of PBT/NBR blends indicates degradation of the plastic resin during blending.
4. Examples of possible effects on the thermoplastic phase are MW reduction, graft formation, and crystallization effects.

Figure 1: Blending Timeline of Nitrile Rubbers

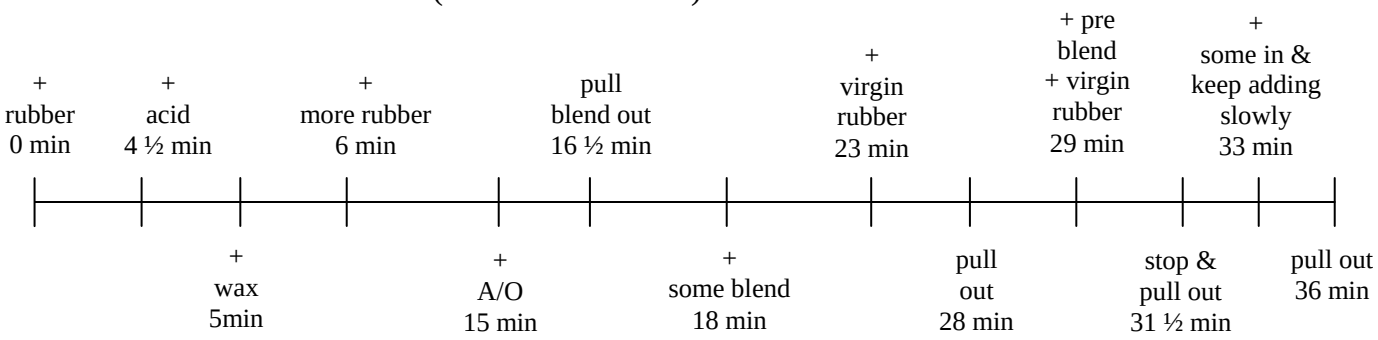
Rubber for PA/NBR Blend (040130-3 Sartomer + Peroxide w/o MgO)



Rubber for PP/NBR Blend (040414-1 Sartomer + Magnesium w/o Peroxide)



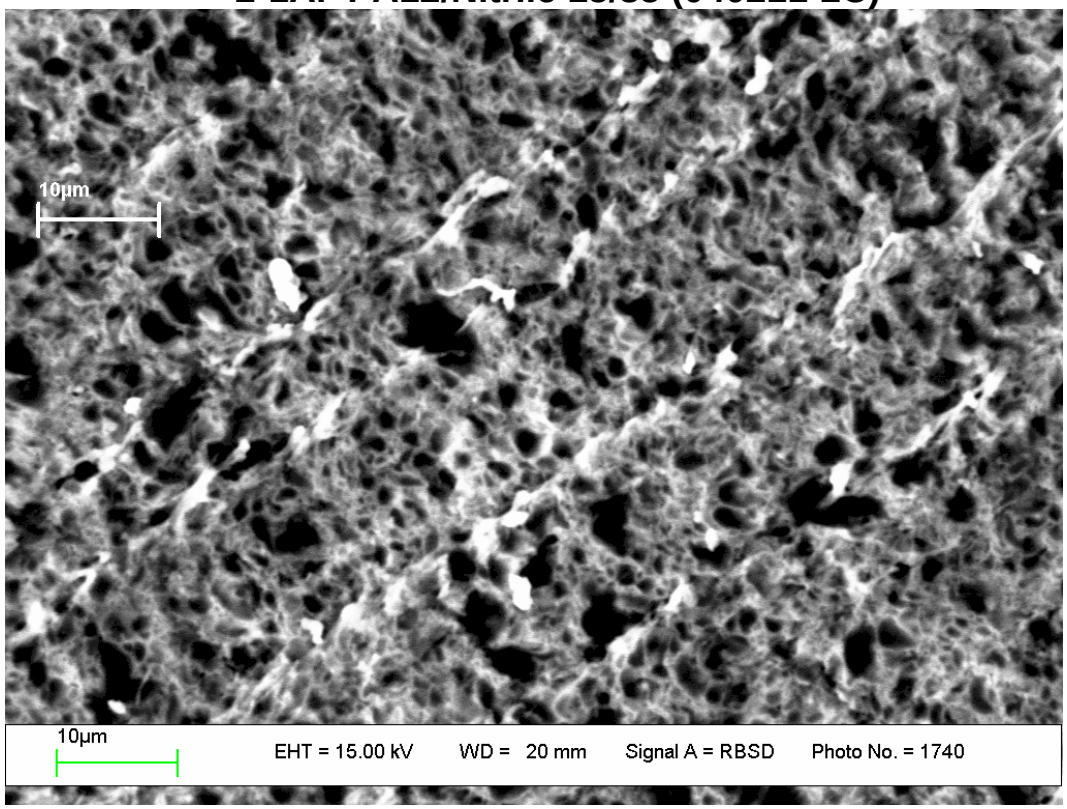
Rubber for PBT/NBR Blend* (040129-1 Sartomer)



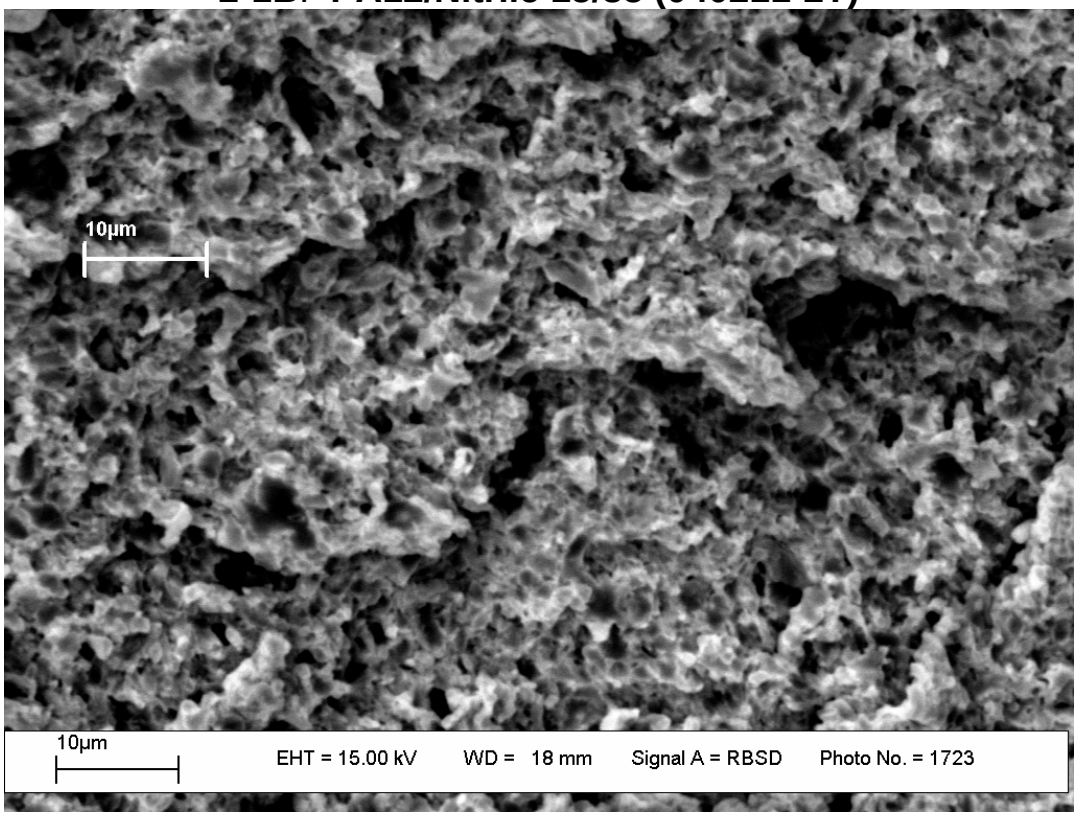
*Timeline is a previous blend 040128-1 then continued next day and finished as 040129-1

Figure 2: Scanning Electron Microscopy of selected thermoplastic rubber blends with thermoplastic resin content from 15-40%. Samples were stained with osmium tetraoxide and the rubber phase containing nitrile rubber is lighter.

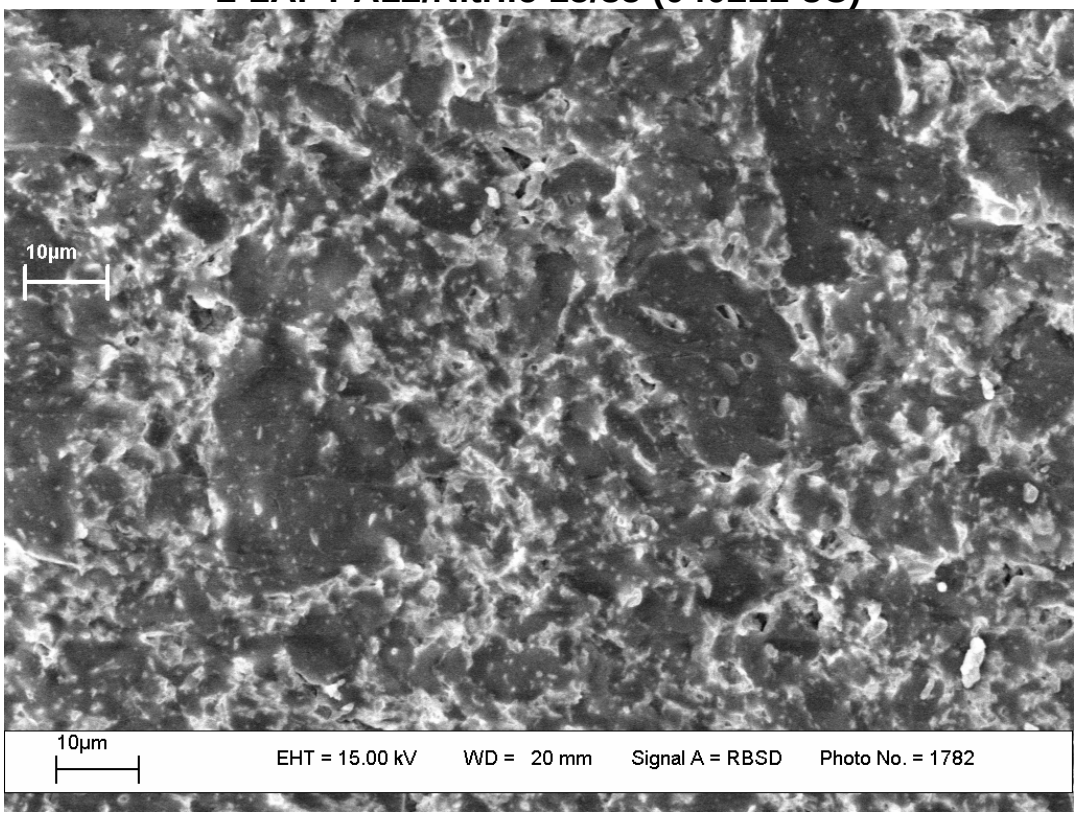
2-1A. PA12/Nitrile 15/85 (040211-2C)



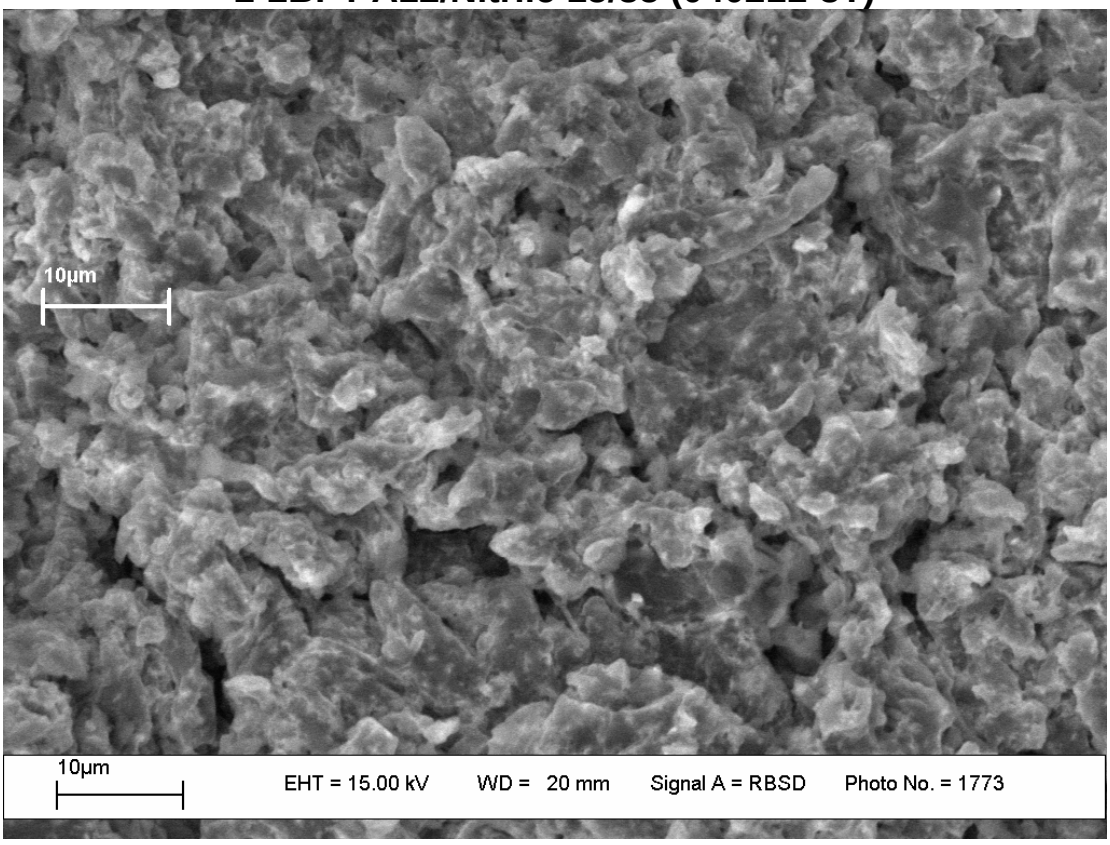
2-1B. PA12/Nitrile 15/85 (040211-2T)



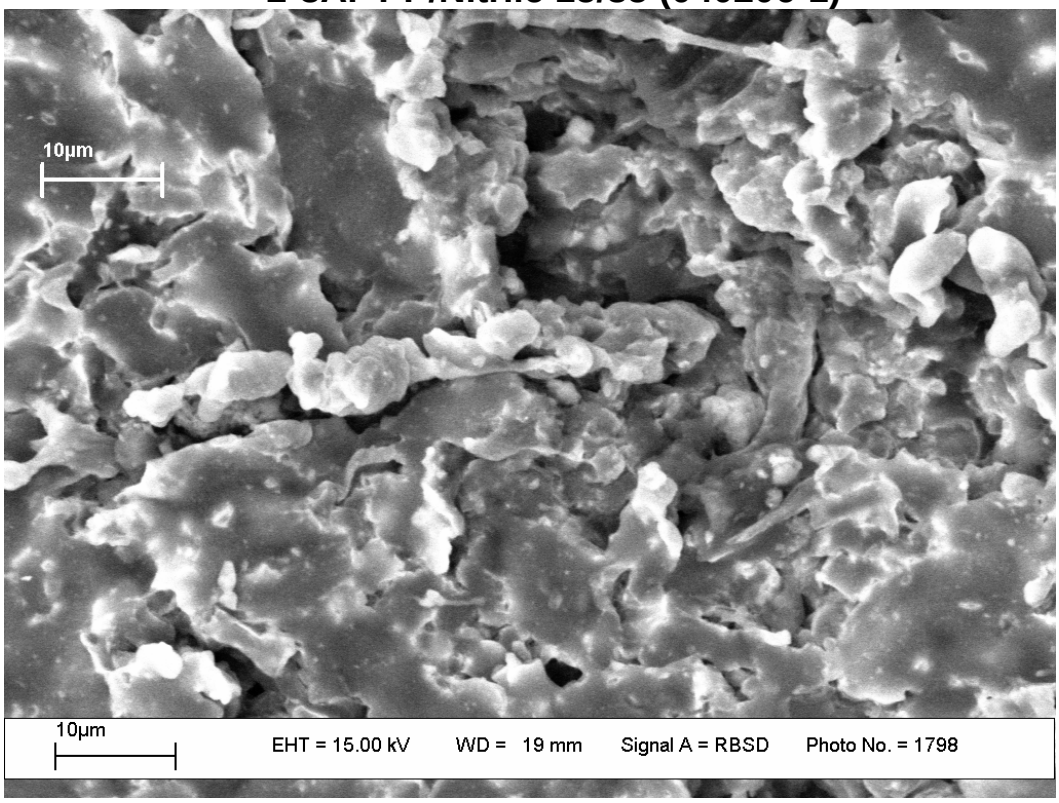
2-2A. PA12/Nitrile 15/85 (040211-3C)



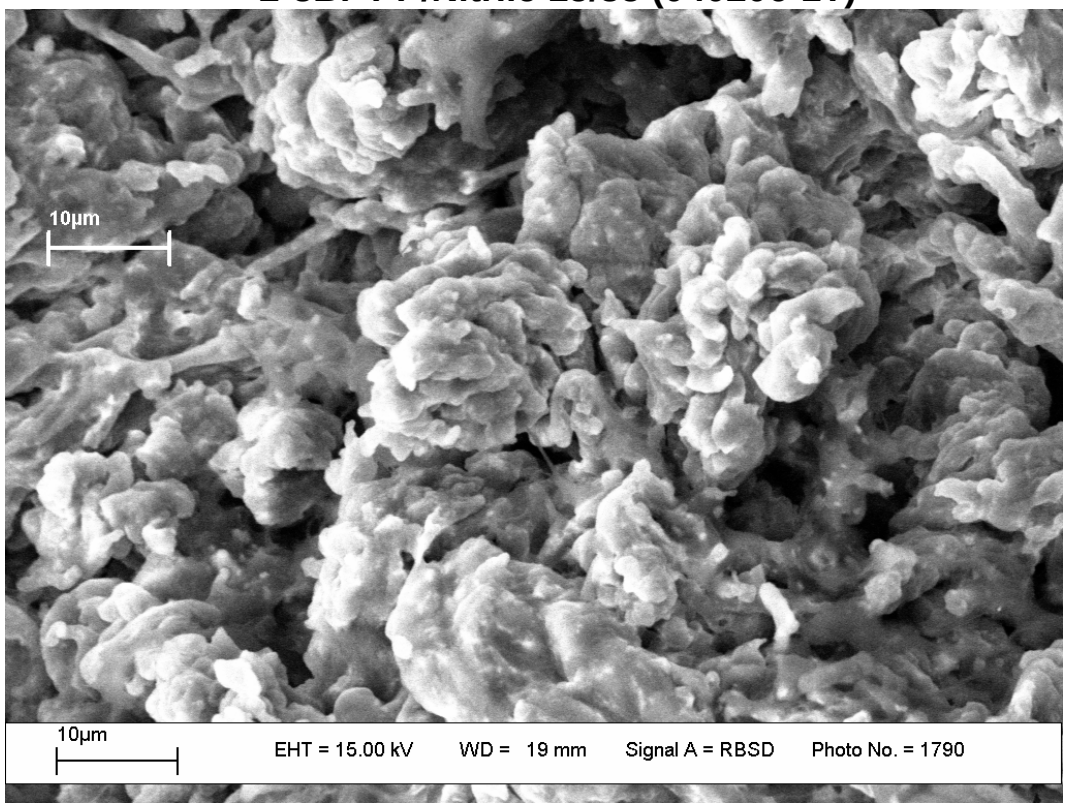
2-2B. PA12/Nitrile 15/85 (040211-3T)



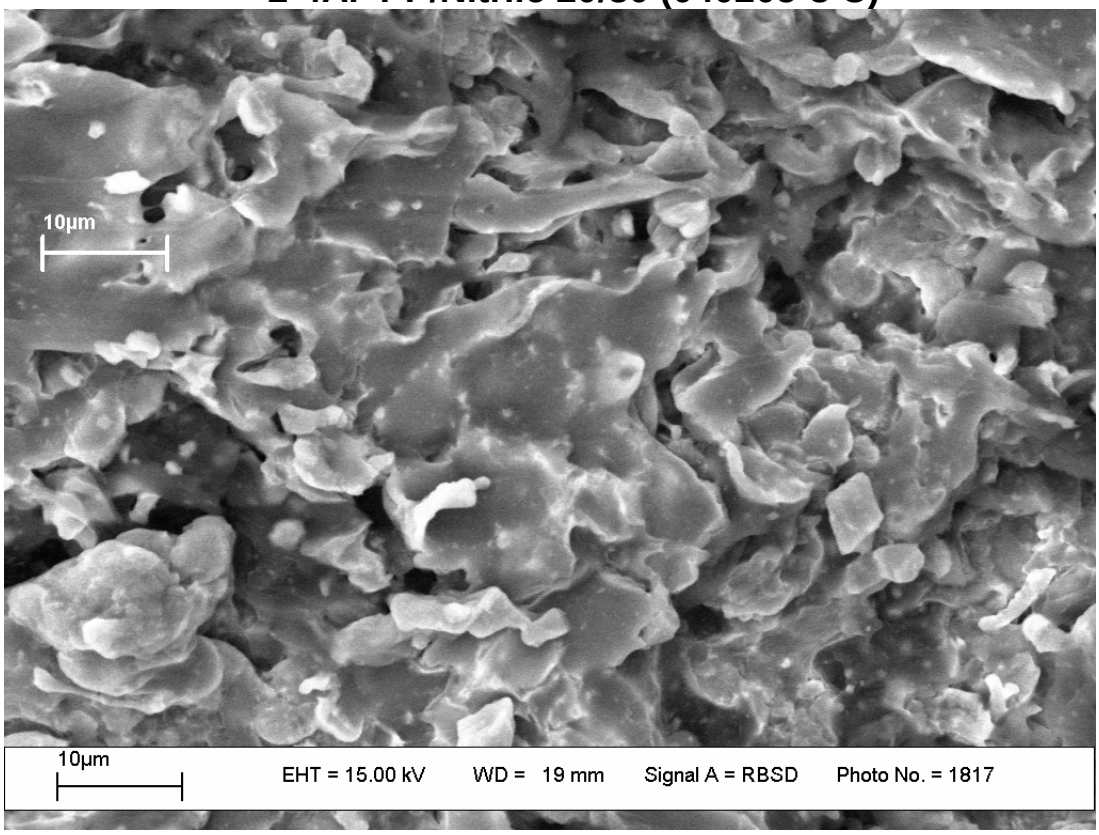
2-3A. PP/Nitrile 15/85 (040206-1)



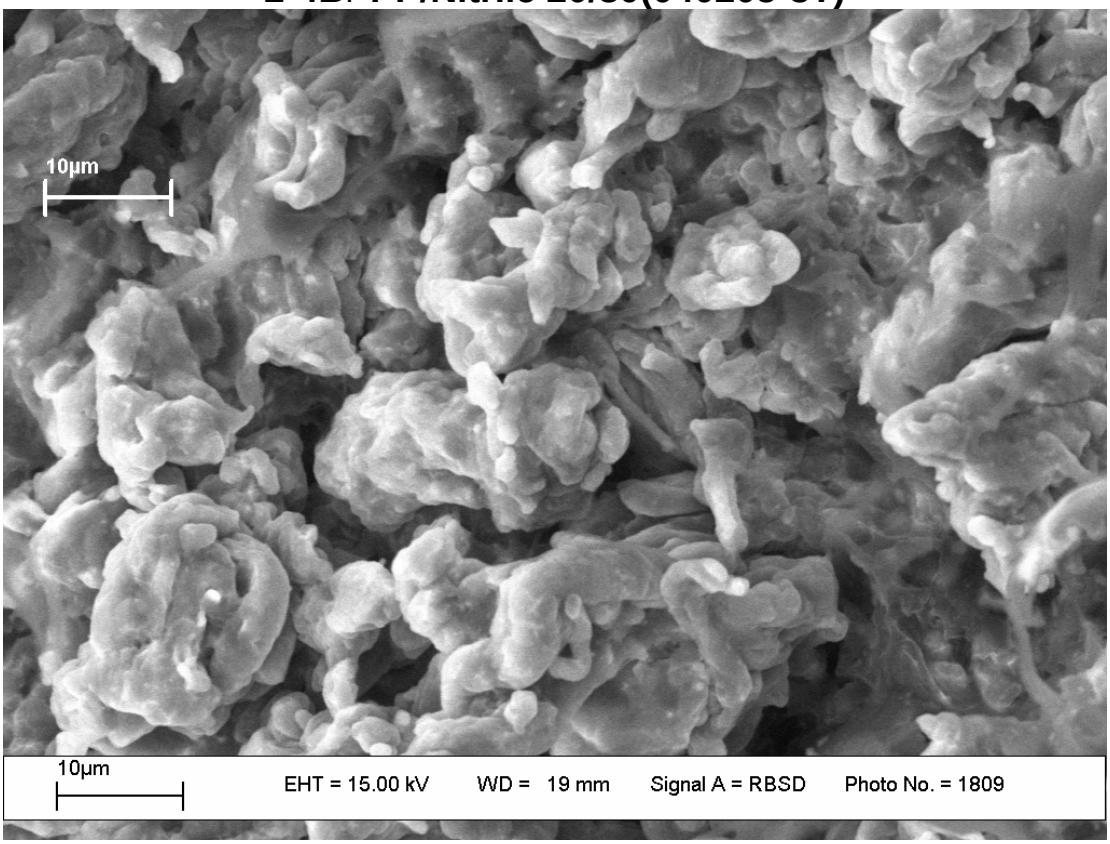
2-3B. PP/Nitrile 15/85 (040206-1T)



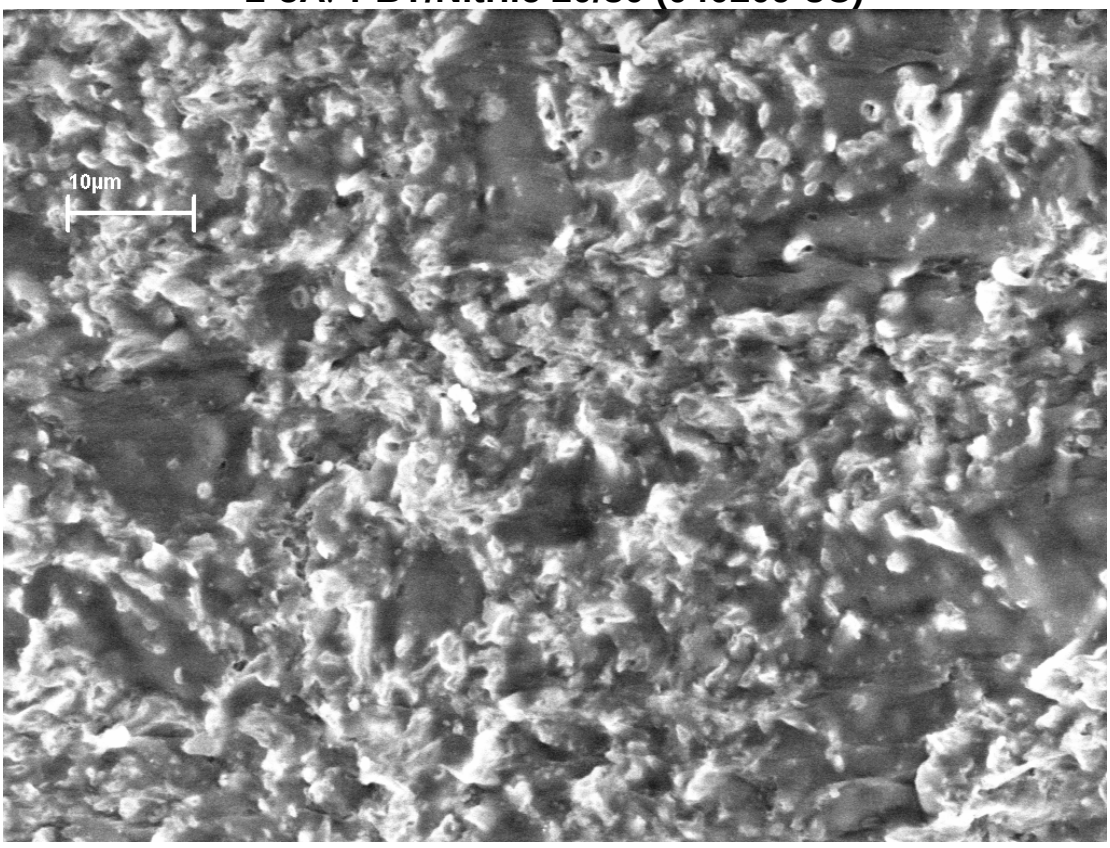
2-4A. PP/Nitrile 20/80 (040205-3 C)



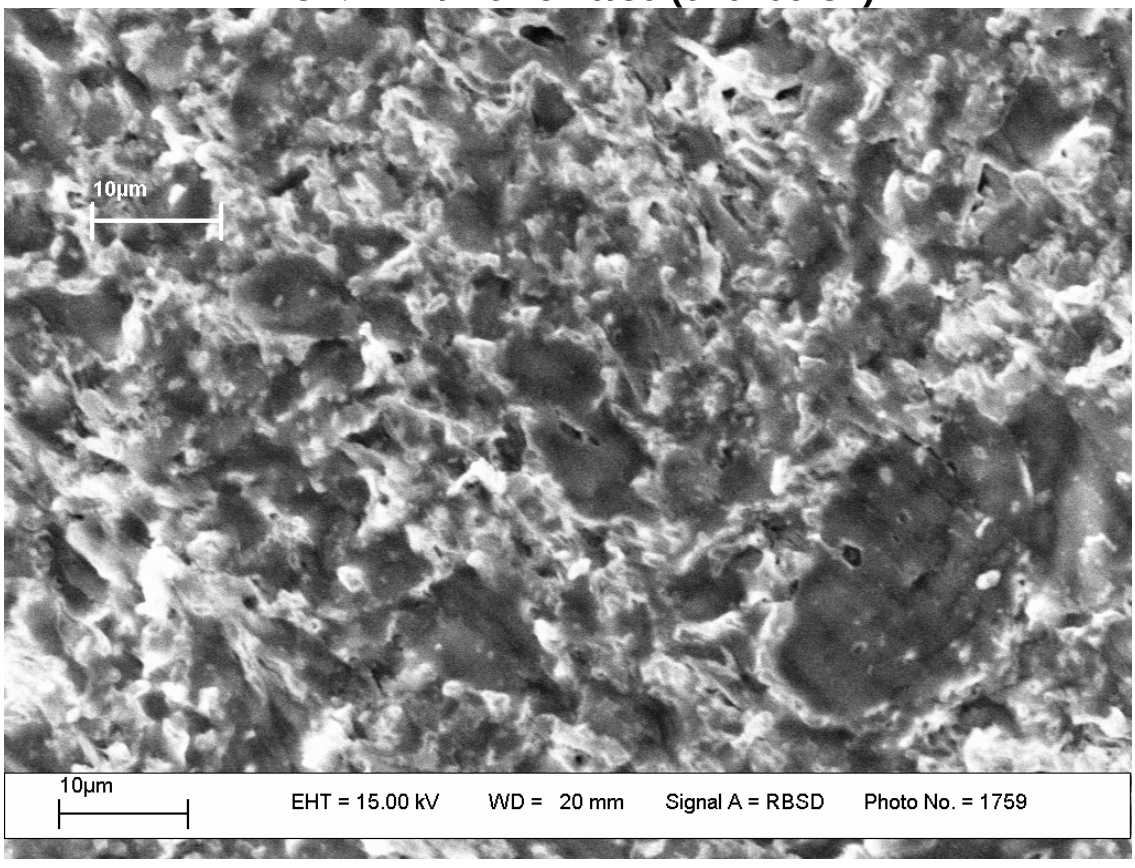
2-4B. PP/Nitrile 20/80(040205-3T)



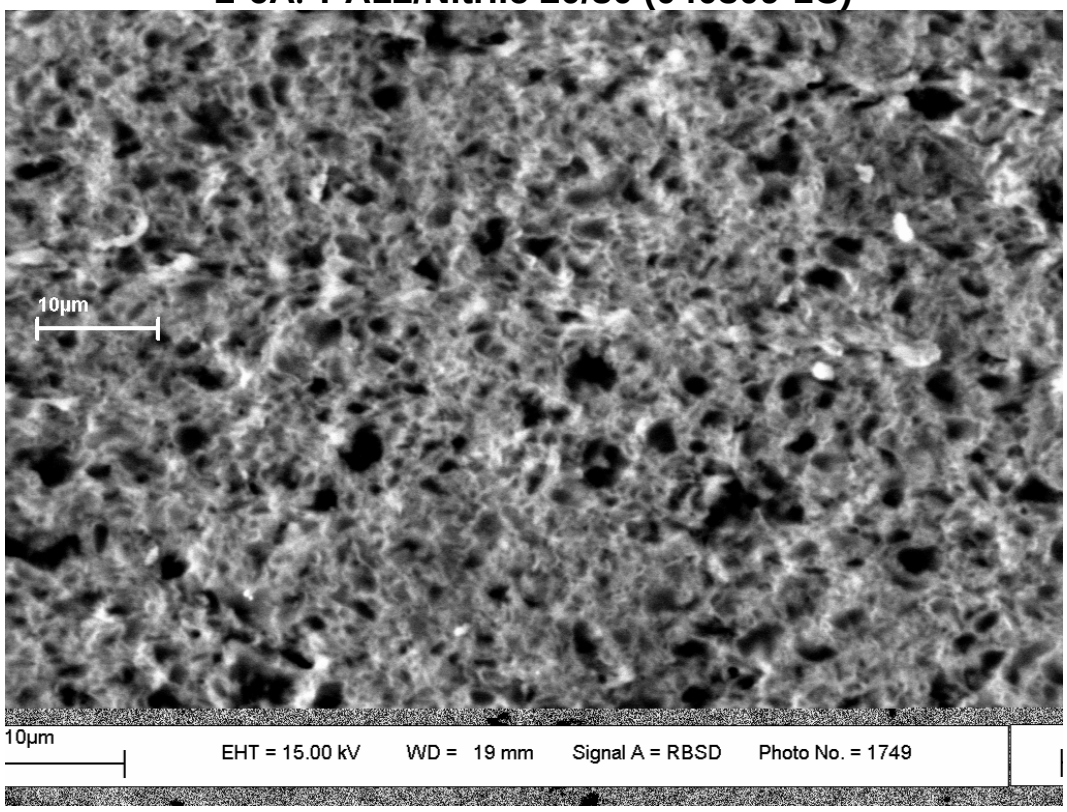
2-5A. PBT/Nitrile 20/80 (040209-3C)



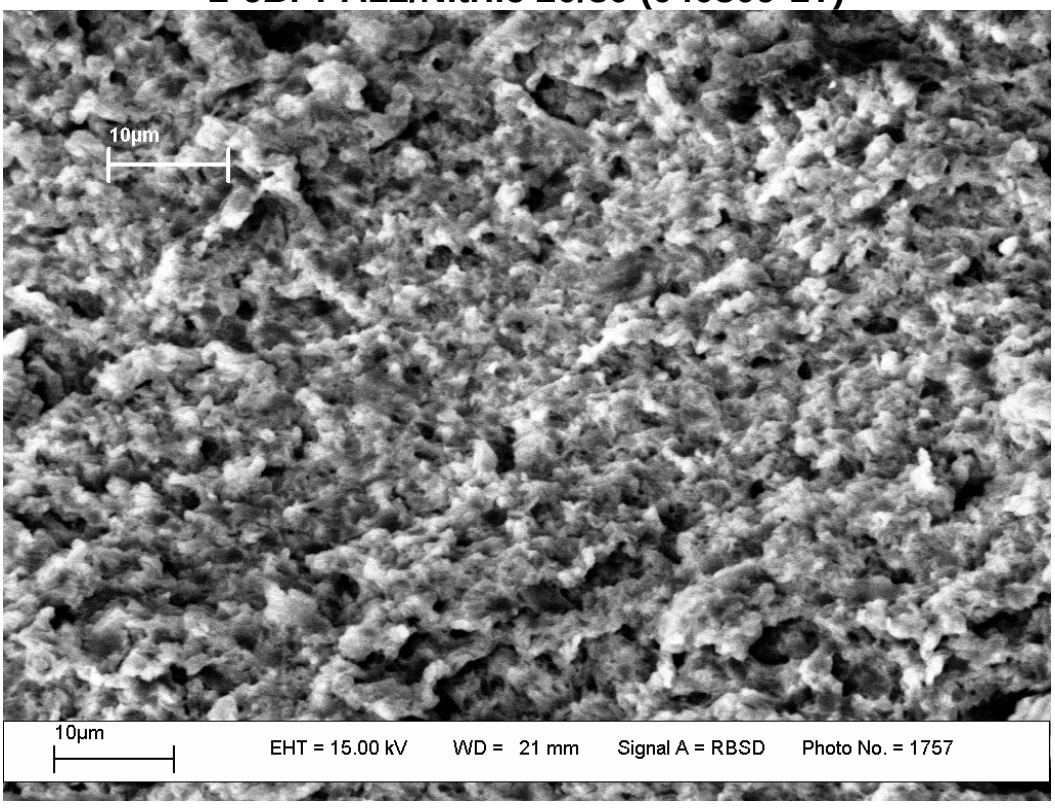
2-5B. PBT/Nitrile 20/80 (040209-3T)



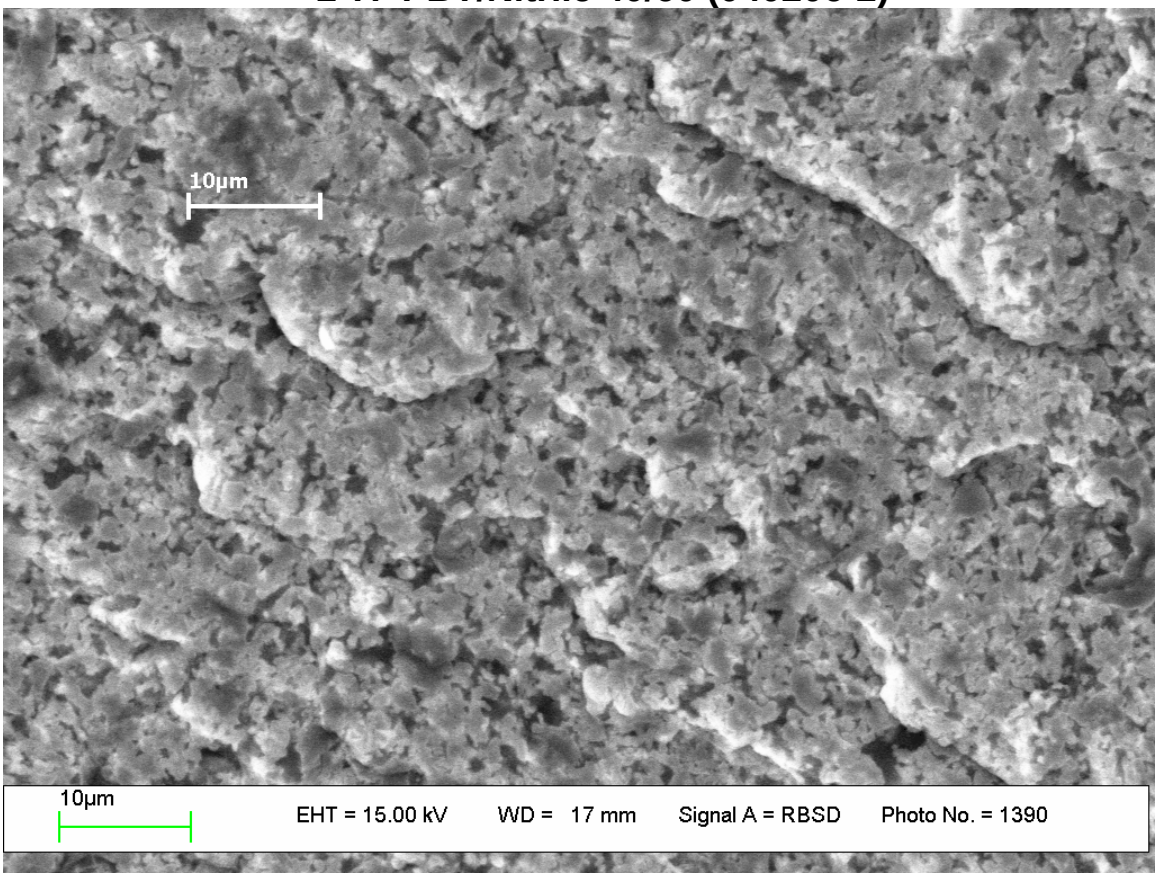
2-6A. PA12/Nitrile 20/80 (040309-1C)



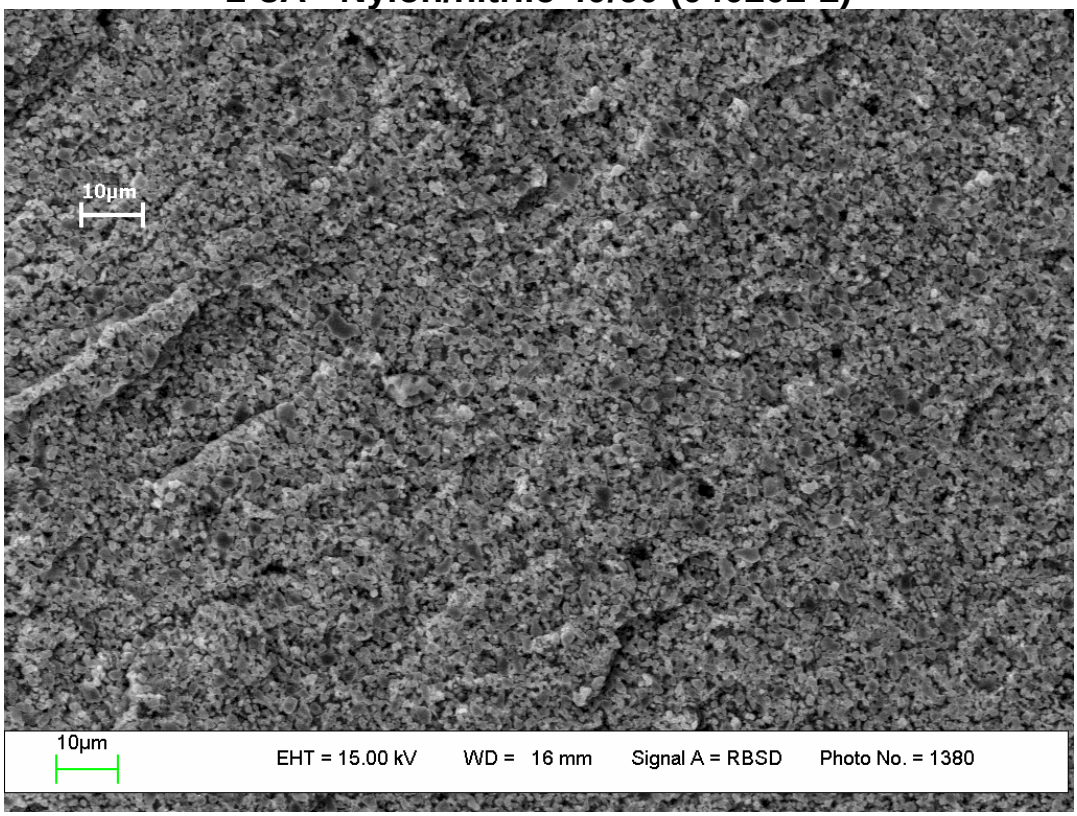
2-6B. PA12/Nitrile 20/80 (040309-1T)



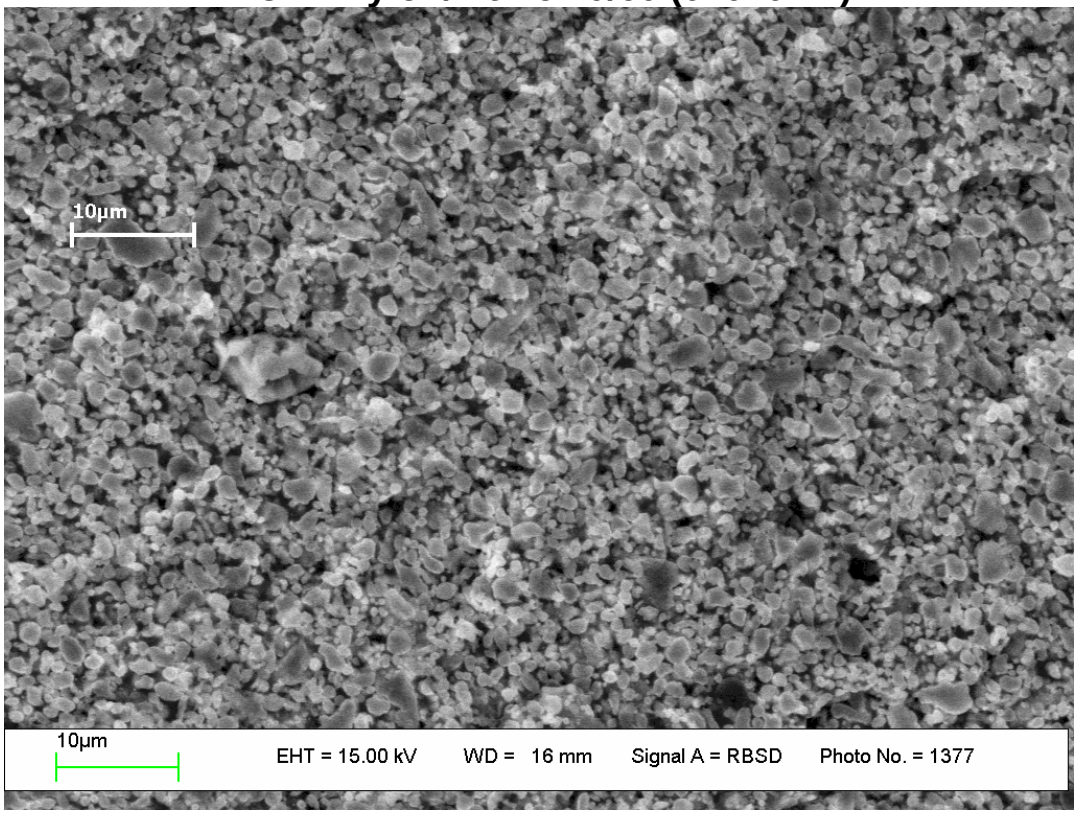
2-7. PBT/Nitrile 40/60 (040206-2)



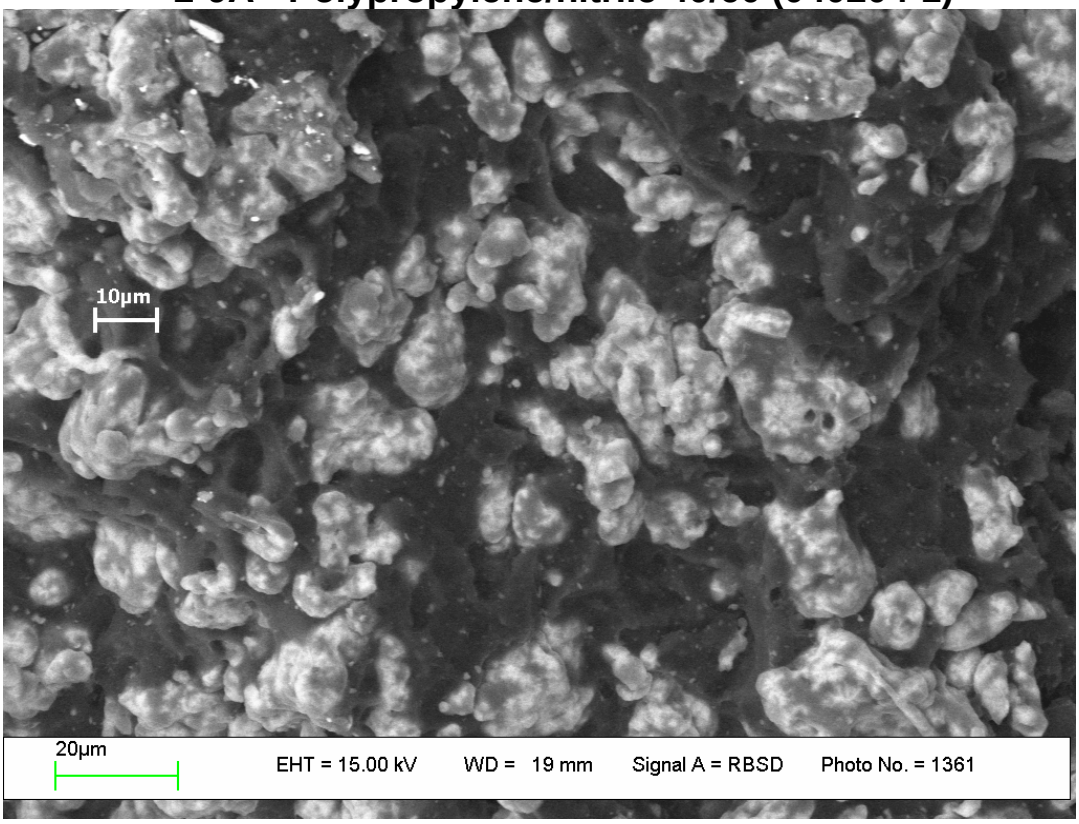
2-8A - Nylon/nitrile 40/60 (040202-2)



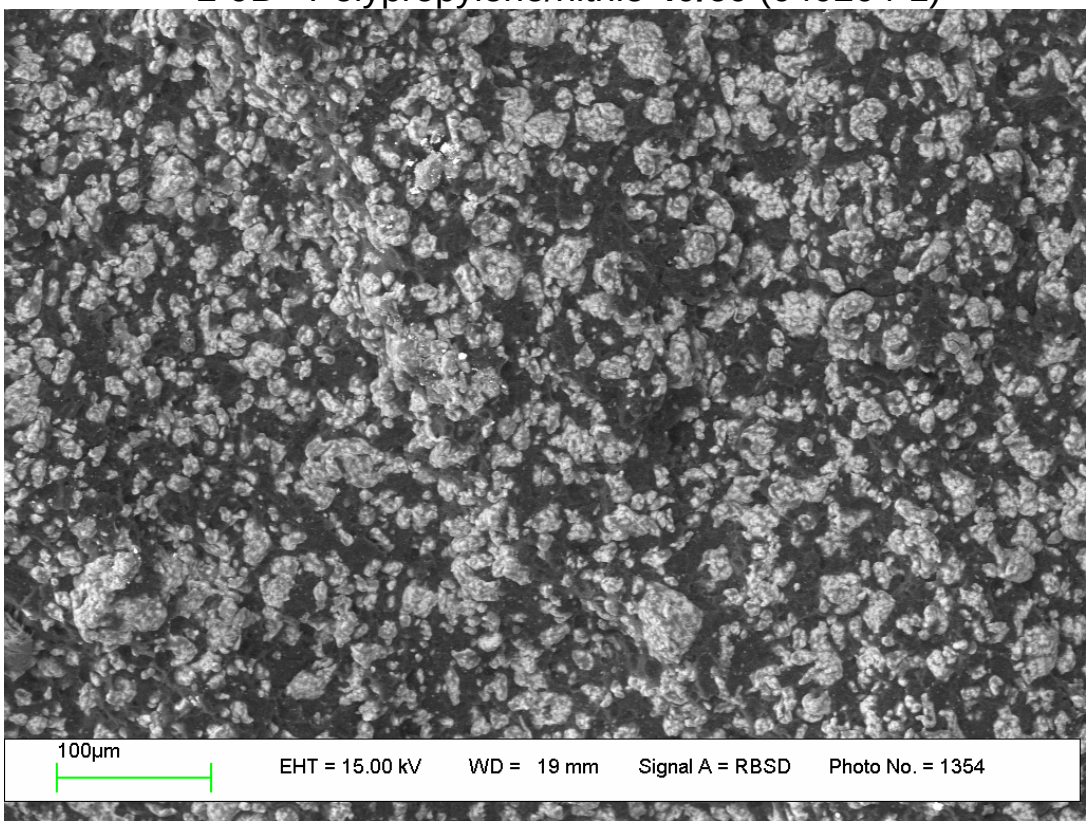
2-8B - Nylon/nitrile 40/60 (040202-2)



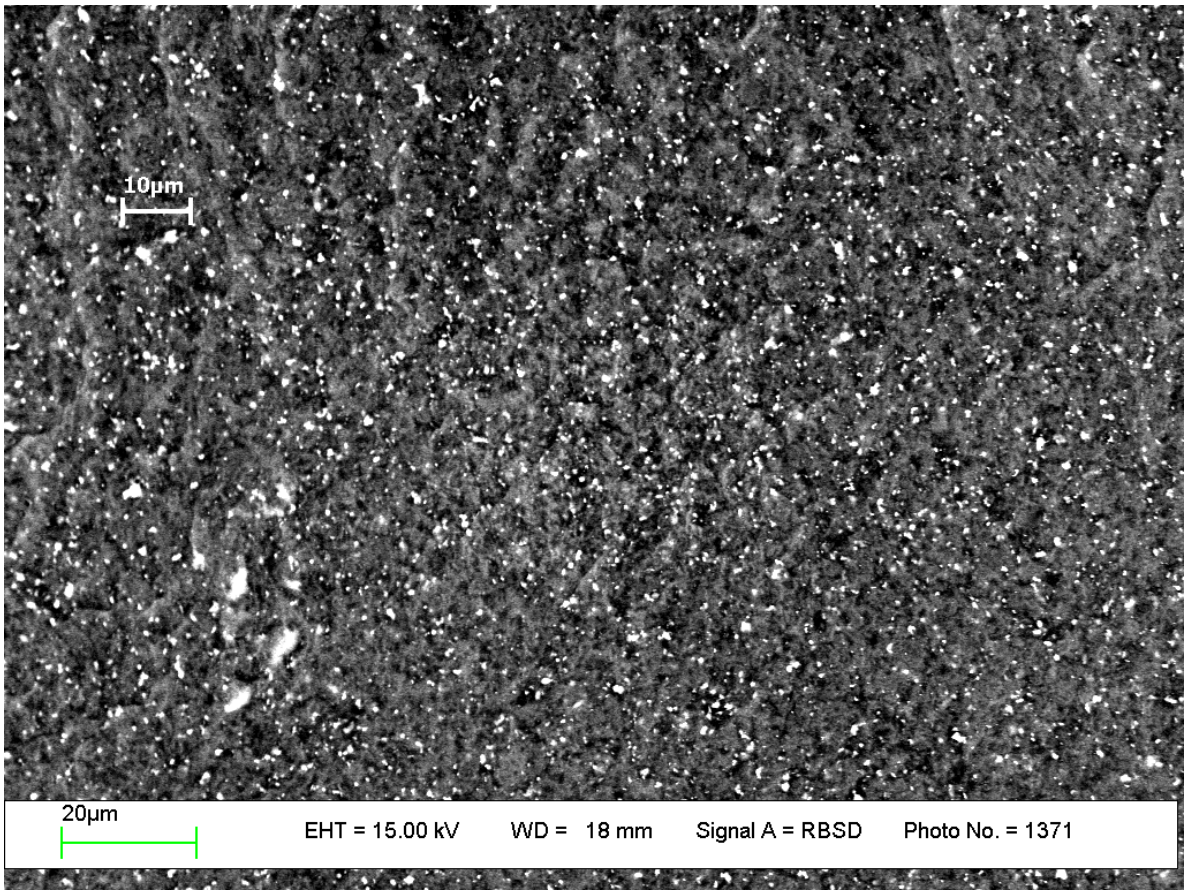
2-9A - Polypropylene/nitrile 40/60 (040204-1)



2-9B - Polypropylene/nitrile **40/60** (040204-1)



2-10 - Polypropylene/chlorobutyl 40/60 (040126-1)



OVERALL CONCLUSIONS:

1. Dynamic vulcanization can produce a variety of rubber plastic blends, many with elastomeric properties.
2. Both rubber and plastic phases are affected during the dynamic vulcanization process.
3. Under exposure to solvents both rubber and rubber/plastic blends undergo a rapid approach to equilibrium swelling.
4. Swelling index values of blends are significantly less than the expected "theoretical" values, based strictly on composition in the blend. This is attributed to a "caging effect" of the thermoplastic phase on the rubber phase at higher thermoplastic compositions.
5. In blends of rubber and thermoplastic, minimum elongation values are reached at phase inversion.
6. Increased compatibility in blends can be correlated with reduced particle size in the discrete phase. As well, increased compatibility frequently produces less caging effect on the rubber phase.
7. With respect to re-emission values for the five blends systems studied, from best to worst, the order is PA/CIIR > PP/CIIR > PA/NBR > PP/NBR > PBT/NBR

APPENDIX

"Solvent Resistance and Mechanical Properties in Thermoplastic Elasotomer Blends Prepared by Dynamic Vulcanization" presented during the IUPAC International Polymer Congress in Paris (July 4-8, 2004).

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The report presents work on five types of dynamically vulcanized thermoplastic rubber blends made from chlorobutyl and nitrile rubbers and polypropylene, nylon 12, and PPT resins.

The project objective was to establish a correlation between blend composition and properties such as microstructure swelling index, tensile strength, elongation at break, hardness, and resistance to penetration/reemission of chemical warfare agents.

The method of blend preparation and materials testing was described.

Results were reviewed and conclusions were drawn.

The results confirm that dynamic vulcanization can produce a variety of rubber plastic blends, many with elastomeric properties. Both rubber and plastic phases seems to be affected during the dynamic vulcanization process. Under exposure to solvents, both vulcanized rubber and rubber/plastic blends underwent a rapid approach to equilibrium swelling. It was found that swelling index values of blends were significantly less that the expected “theoretical” values, based strictly on composition in the blend. This was attributed to a caging effect of the thermoplastic phase on the rubber phase at the higher thermoplastic composition.

In blends of rubber and thermoplastic, the minimum elongation at break seems to be reached at phase inversion. With respect to re-emission values for the five blends systems studied, from best to worst, the order is PA/CIIR > PP/CIIR > PA/NBR > PP/NBR > PBT/NBR.

Selected results were presented during the IUPAC International Polymer Congress in Paris (July 4-8, 2004). Overheads from the presentation are enclosed with report.

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Polybutyleneterephthalate, polypropylene, polyamide 12, chlorobutyl rubber, nitrile rubber, dynamic vulcanization, swelling index, microstructure, tensile strength, elongation at break, hardness, resistance to penetration, reemission